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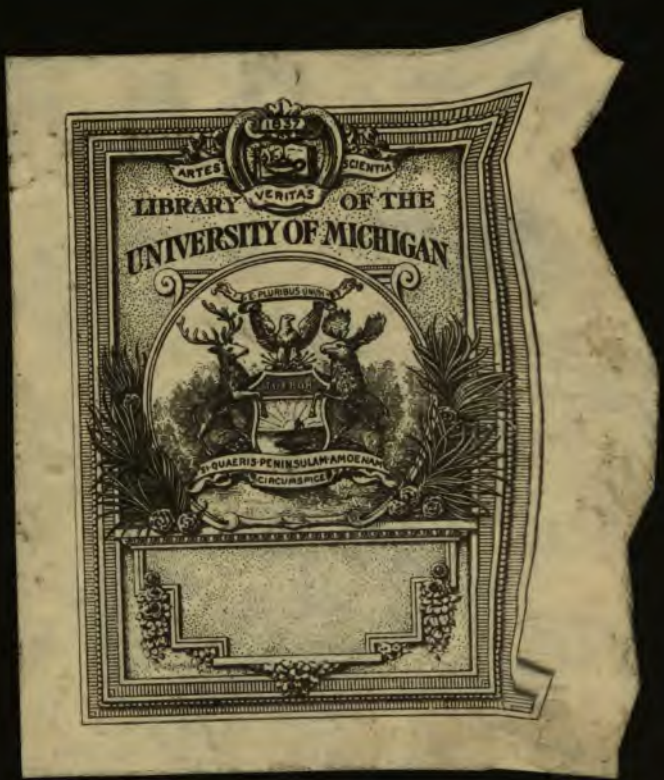
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OF

DAIRY ANALYSIS

BY

H. DROOP RICHMOND, F.I.C.

ANALYST TO THE AYLESBURY DAIRY COMPANY, LIMITED

ILLUSTRATED WITH PHOTOGRAPHS BY THE AUTHOR

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CHARLES GRIFFIN AND CO. LIMITED

EXETER STREET, STRAND

1905

PREFACE

THIS work is intended to contain working directions for the analysis of milk and dairy-products; the estimation of all constituents of diagnostic value is shortly described in detail, and is in many cases illustrated by photographs of chemists actually carrying out the determination.

A chapter on the application of analysis to the solution of problems usually placed before the chemist is included, and a very short summary of the composition of milk and its products is given.

In the Appendix the composition and preparation of the various solutions is detailed. Tables are given to facilitate the working out of results; these Tables are condensed to occupy one page each, and the saving of time by avoiding the turning over of pages will more than compensate for the slight extra labour due to the condensation.

While not intended to be a complete guide to the analysis of milk, it is hoped that this work will afford assistance to analysts, health officers, dairy students, and those engaged in the supervision of dairies; with this object in view the more simple tests have been described in a manner which will render their working by

208754

persons other than chemists possible; it must be remembered, however, that though these methods are easy, they are often fallaciously easy, and lack of chemical training may lead to the making of errors, and the overlooking of important points; no amount of careful following of directions can replace a thorough training in chemical science and manipulation, and though simple tests have a real value as a guide, they have not the reliability of an analysis made by a skilled chemist.

H. D. R.

September 1905.

CONTENTS

CHAPTER I

INTRODUCTION

PAGE

The Constituents of Milk — Products Derived from Milk—Composition of Milk—and of its Products . 1

CHAPTER II

THE ANALYSIS OF MILK

Preparation of the Sample—Specific Gravity—Total Solids—Ash—Mineral Constituents—Acidity—Fat —Gerber Method — Gravimetric Methods — Milk Sugar — Proteids — Nitrogen — Curd — Relation between Fat, Solids not Fat, and Specific Gravity . 5

CHAPTER III

THE ANALYSIS OF LIQUID MILK-PRODUCTS

Skim Milk — Cream — Butter-milk—Whey — Sterilised Milk—Condensed Milk—Sour Milk 43

CHAPTER IV

THE APPLICATION OF ANALYSIS TO THE SOLUTION OF PROBLEMS

The Detection of Adulteration—Preservatives—Poor Milk—Sweet Milk—High Colour—Sour Milk—Unusual Taste—Dirty Milk—Detection of Adulteration of Cream—and Skim Milk 49

CHAPTER V

The Analysis of Butter

PAGE

Water—Curd and Salt—Preservatives—Examination of the Fat—Reichert-Wollny Method—Soluble and Insoluble Fatty Acids and Mean Molecular Weight—Density—Examination under Polarised Light—Iodine Absorption—Refractive Index—Detection of Adulteration 60

CHAPTER VI

The Analysis of Cheese

Water, Ash, and Salt—Fat—Nitrogen—Products of Ripening—Examination of the Fat—Detection of Adulteration—Application of Analysis to Cheese-making 75

Tables for Calculation 79

Appendix. Standard Solutions 82

Index 87

CHAPTER I.

INTRODUCTION.

Milk consists of (1) fat in small globules (Fig. 1) ranging in size from 0.01 mm. in diameter to 0.0016; (2) milk-sugar and (3) various salts in solution in water; and (4) casein, combined with lime and

FIG. 1.—Milk (magnified 400 diameters).

phosphoric acid, and (5) albumin in less perfect solution. There are in addition (6) other compounds in small quantities.

The fat will be treated of in the section on butter; the milk-sugar belongs to the class of carbo-hydrates and crystallises with 1 OH_2, and is one of the hexa-bioses. It rotates the plane of polarisation, its specific

rotatory power being 52.5° for the crystallised sugar; and reduces solutions of copper salts.

Casein is a proteid belonging to the class of the nucleo-albumins; it contains carbon, hydrogen, oxygen, nitrogen, sulphur, and phosphorus; in milk it exists as a salt of lime and soda combined with calcium phosphate; acids precipitate the free casein if dilute, while strong acids re-dissolve it. Rennet splits casein up

FIG. 2.—Cream (magnified 400 diameters).

into curd, which is a combination of para-casein with the lime and the calcium phosphate of the casein, the soda being split off, and whey proteid which is free from phosphorus.

Albumin is a proteid which is distinguished by coagulating on heating to 70° C.; in milk it probably exists as a salt, and this does not coagulate until the milk is acidified. Unaltered albumin is not precipitated by acids.

When micro-organisms act on milk various products are formed; the most important change is the formation of lactic acid from the sugar, which causes milk to become sour, and curdles it by precipitating the casein.

The fat globules are lighter than the aqueous serum,

and they tend to rise. *Cream* (Fig. 2) is the upper portion of milk after standing, and differs from milk practically only in that it contains more fat and proportionately less serum. *Skim-milk* is the milk deprived of the bulk of its cream, and if the separation of cream has been performed in a centrifugal separator it is practically free from fat and contains only the aqueous serum. This is termed *separated* or *machine-skimmed milk.*

When cream (or milk) is suitably agitated for some time, the fat globules coalesce to small granules, and these after working together into a nearly homogeneous mass form *butter.* This is chiefly composed of fat, but contains some water and other constituents of milk. The residue is termed *butter-milk,* which does not differ greatly from skim-milk in composition.

By treating milk with rennet, *curd* is separated; this carries down the bulk of the fat, and after pressing, salting, and ripening, partly by the action of micro-organisms and partly by the action of the natural ferments of milk, it is converted into *cheese;* cheese consists essentially of fat, para-casein, and products derived from the latter together with some water and salts.

The following table gives the average morning and evening milk for each month, and represents the percentage composition during 1904:

TABLE I.

	Morning Milk.			Evening Milk.		
	Total Solids.	Fat.	Solids not Fat.	Total Solids.	Fat.	Solids not Fat.
January .	12.58	3.61	8.97	12.96	3.98	8.98
February .	12.54	3.57	8.97	12.86	3.89	8.97
March . .	12.52	3.55	8.97	12.82	3.86	8.96
April . .	12.36	3.45	8.91	12.81	3.91	8.90
May . . .	12.24	3.28	8.96	12.85	3.87	8.98
June . .	12.25	3.29	8.96	12.75	3.81	8.94
July . . .	12.26	3.42	8.84	12.64	3.87	8.77
August . .	12.33	3.48	8.85	12.77	3.96	8.81
September	12.50	3.56	8.94	12.96	4.03	8.93
October .	12.60	3.63	8.97	13.07	4.11	8.96
November .	12.72	3.70	9.02	13.09	4.11	8.98
December .	12.76	3.71	9.05	13.08	4.07	9.01

In the Table below is given the average percentage composition of milk and the various products derived from it.

TABLE II.

	Water.	Fat.	Milk sugar.	Proteids.	Mineral Matter.
Milk	**87.20**	**3.80**	**4.75**	**3.40**	**0.75**
Separated Milk	90.40	0.20	4.95	3.57	0.78
Thick Cream .	39.37	56.09	2.29	1.57	0.38
Thin Cream .	67.50	25.67	3.66	2.60	0.57
Fresh Butter .	12.99	85.81	0.37	0.74	0.09
Salt Butter .	13.78	82.97	0.39	0.84	2.02*
Butter-milk .	90.33	0.76	4.78‡	3.33	0.80
Whey . . .	93.21	0.30	4.99	0.92	0.58
Curd	49.43	27.38	2.04	20.00	1.15
Cream Cheese .	30.66	62.99	0.26‡	4.94†	1.15
Soft Cheese .	50.04	27.50	?	18.32†	4.12*
Hard Cheese .	33.89	33.00	1.90‡	27.56†	3.65*
Half-skim Cheese	37.35	24.61	?	32.40†	5.65*

* Including added salt.
† Including products of ripening.
‡ Including lactic acid.

CHAPTER II.

THE ANALYSIS OF MILK.

Preparation of the Sample.—A milk sample conveniently consists of a four-ounce bottle filled nearly full; if the sample is to be representative of a bulk, the milk (whether in a churn, pail, can or jug) should invariably be well stirred before the sample is taken in order to distribute the cream which always tends to rise to the surface. Samples taken in a dairy or other place for testing on the premises may, however, be taken in cans, and if the sample is one frequently taken from the same source a distinguishing mark may be stamped on the can.

On receipt of the sample in the laboratory it should invariably be stirred before any portion is withdrawn for analysis; violent shaking is to be deprecated, as not only is there a tendency to churn the fat, but air bubbles, which do not separate immediately, are included, and prevent accurate measurements, especially of specific gravity. In cold weather samples are often frothy, and while they remain cold the air bubbles separate very slowly. Freshly drawn milk is also frothy, and the fat being in the liquid condition has a lower specific gravity than it has after solidification. If the sample is turning sour there is often difficulty in uniformly distributing the cream, and violent shaking may have to be resorted to; if any of the fat is churned or becomes churned in this operation, the lumps of churned fat should be removed, dried, and the fat extracted with ether and weighed; the total weight of milk is ascertained, and the percentage of churned fat

calculated. The remainder of the sample is analysed separately.

Milk which is sour and curdled is mixed by turning

FIG. 3.—Putting Lactometer into Milk.

the whole sample into a beaker, and whipping with a brush made of fine wires.

Always determine the specific gravity of every sample if possible.

Estimation of Specific Gravity by Lactometer.—Nearly fill a cylindrical vessel of depth such

that the lactometer shall float, and at least ¼ inch wider than the lactometer; a glass jar, or tin pot, or even a milk-can serves well. Hold the lactometer at an angle (Fig. 3), and cautiously lower it into the milk,

FIG. 4.—Vieth's Lactometer.

taking care that no air bubbles are retained in the space between the upper and lower bulbs (see Fig. 4); when the upper bulb is partially below the surface, raise to an upright position and immerse the lactometer to the 30° mark, and let it find its own level. When steady read off

the point where the surface of the milk cuts the stem; this point is not visible, as the milk is drawn up round the stem by capillary attraction (Fig. 5), and must be mentally estimated. Some little practice is required to do this, and the reading may be obtained by observing the point on the stem to which the milk reaches, and adding a constant amount for the height of the meniscus, usually ½ degree; thus in the figure the true reading of the lactometer is 32.5°, the apparent reading is 32°, and with ½ degree added on the true reading is obtained.

FIG. 5.—Lactometer in Milk.

The jar in the figure has been filled to the brim with milk in order to show clearly the effect of capillary attraction; it is neither necessary nor advisable to do this when testing milk.

Immerse the bulb of a thermometer in the milk, and stir the milk till the temperature is constant; correct the reading to 60° F. by means of the table given on p. 79 or by the milk scale (p. 40). To use the table, find in the top line column the specific gravity (or the nearest figure to the left), and in the left-hand column the temperature; where the lines intersect the corrected specific gravity is given (adding on, if the specific gravity found is not an exact degree, the decimals).

A thermo-lactometer, *i.e.*, a lactometer which contains a thermometer, may be used, and the temperature can then be read off at the same time as the specific gravity (Fig. 6).

Never take a specific gravity reading without also noting the temperature and correcting to 60° F.

The lactometer should be checked by the gra-

vimetric method, to make sure that the scale is correct.

A lactometer does not give accurate results if a film of milk is allowed to dry on it; if a sample has been

FIG. 6.—Thermo-lactometer.

tested and the lactometer removed, and allowed to stand, it must be washed and dried before being used for another sample.

There is usually little objection, however, to removing a lactometer from one sample and, after draining, placing it at once in another sample; but if the sample

is likely to be the subject of legal proceedings or is otherwise important, the lactometer should always be cleaned before testing.

The Gravimetric Estimation of Specific Gravity.—Dry a Sprengel tube (Fig. 7) by washing

FIG. 7.—Sprengel Tube.

with distilled water, alcohol, and ether, and placing in the water-oven; aspirate air through when still hot, cool and weigh. Fill the tube with distilled water, and place it in a vessel containing water at 15.5° C. till the

water inside no longer alters in volume; adjust the level of the water accurately to the mark on the wider tube by cautiously applying filter paper to the narrower end; wipe the outside of the tube dry, and weigh. The difference between the weight of the full and the empty tube gives its capacity in grammes of water at 15.5°.

Empty the water, and rinse the tube several times with the milk, and then fill it with milk; allow the tube to stand a minute to permit air bubbles to rise, suck or blow these out, and fill the tube completely. Immerse the tube in water at 15.5° C. till the volume ceases to alter, and then wipe dry and weigh as before. The difference between the full and the empty tube gives the weight of milk, and this divided by the capacity in grammes of water at 15.5° gives the specific gravity at 15.5°.

The capacity of the tube once determined remains constant, and it need not therefore be determined every time; the weight of the empty tube should, however, be taken occasionally.

Rise of Specific Gravity of Milk on Standing.—If the milk is freshly drawn or has been recently heated, the fat is in the liquid condition, and its specific gravity is lower than when solid. The solidification of the fat globules takes some time, and twelve to twenty-four hours may elapse before the maximum specific gravity is attained. If the milk is frothy, air bubbles may cause the specific gravity to appear too low.

The maximum specific gravity is taken as the correct figure.

Estimation of Total Solids.—Weigh a basin either of platinum or of porcelain, preferably 2¾ in. wide and flat-bottomed; pipette in 5c.c. of milk, and weigh again; the weighing should be rapid, but the exactitude of weighing need not be more than 2mg. Place the basin on a water-bath (Fig. 8), and from time to time break the skin with a needle; when

apparently dry place in a water-oven (Fig. 9), and continue the drying for four hours, cool in a desiccator, and weigh; replace in the oven for periods of one hour each, cool and weigh, until the loss in one hour is less than 1mg. The weight of the residue divided by the weight of milk taken and multiplied by 100 gives the percentage of total solids.

FIG. 8.—Water-bath.

(*Babcock's method*) The basin may be loosely packed with ignited asbestos, if great accuracy is required; (*Stokes' method*) if speed is wanted, a few drops of a 10 per cent. solution of acetic acid in alcohol may be added, and the time of drying can then be curtailed.

Estimation of Ash.—The total solids are ignited in a muffle furnace (Fig. 10), which should not be allowed to become hotter than a dull red heat; to facilitate burning, upstanding portions of the ash may be broken down by touching with a platinum wire; when all the carbon is burnt away the basin is cooled in a desiccator and weighed. The ignition may be performed over a Bunsen burner (Fig. 11), the point of the flame of which should barely be allowed to touch the bottom of the basin; the basin should be covered with a platinum lid. It is very important not to heat the ash to too high a temperature.

FIG. 9.—Water-oven.

Estimation of Soluble and Insoluble Ash.—Fill the basin with hot water and filter through a

FIG. 10.—Muffle.

small ash-free filter; wash with hot water. Place the filter and its contents in the basin, and ignite; the residue is the insoluble ash and the soluble ash can be obtained by difference.

Estimation of Alkalinity, Chlorine, Lime, and Phosphoric Acid.—To the filtrate containing

the soluble ash add a drop or two of phenolphthalein solution, and titrate with $\frac{N}{10}$ sulphuric acid (see Appendix) till the colour is discharged. Each cubic

FIG. 11.—Tripod, Bunsen and Desiccator.

centimetre of acid indicates 0·0044 gramme CO_2 as carbonates. [*Note.*—The alkalinity is not all due to carbonates, a little phosphate is present.]

To the solution add a few drops of potassium chromate, and titrate with $\frac{N}{10}$ silver nitrate solution

(see Appendix) till a red colour just appears. Each cubic centimetre of silver solution indicates 0.00355 gramme of chlorine. [*Note.*—A little of the chlorine, about 0.01 per cent., is lost on ignition of the total solids.]

For the estimation of lime and phosphoric acid, it is advisable to take another quantity of milk (10c.c. or 25c.c. being preferable); this is dried on the water-bath, ignited, the ash dissolved in a little hydrochloric acid, and the solution boiled; after cooling slightly, ammonia is added drop by drop till a permanent turbidity appears, and just sufficient hydrochloric acid added to remove this. The solution is just brought to the boiling-point, and a saturated solution of ammonium oxalate added drop by drop, so long as a precipitate appears; the solution is kept hot (in a water-oven) for at least two hours, and filtered through a small ash-free filter; the precipitate is transferred to the filter, washed with hot water, and the filter placed in a tared basin, and ignited over a small flame; when the filter paper is all burnt away, the precipitate is moistened with a solution of ammonium carbonate, dried, and very gently ignited. The precipitate, now converted into calcium carbonate, is weighed, and the weight of lime found by multiplying by 0.56; it is usually slightly grey, and contains traces of iron, which are small enough to be neglected.

To the filtrate is added 5 or 10c.c. of magnesia mixture (see Appendix), and about one-tenth its volume of strong ammonia; and after stirring well the liquid is allowed to stand at least 12 hours; it is then filtered through an ash-free filter, the precipitate washed with dilute ammonia, and transferred to the filter, and well washed; the filter is placed in a weighed basin, and ignited at first gently, and finally very strongly till white; the residue of magnesium pyrophosphate is weighed, and the amount of phosphoric acid (as P_2O_5) found by multiplying by 0.6396.

For the determination of other mineral constituents, works on mineral analysis should be consulted.

The Estimation of Acidity.—Place 10c.c. of milk in a white porcelain basin (Fig. 12), add a few drops of phenolphthalein solution, and run in from a

FIG. 12.—Estimation of Acidity.

burette $\frac{N}{10}$ caustic soda solution (see Appendix) in drops, stirring constantly, till a faint pink colour, which does not disappear on stirring, is produced; each $\frac{1}{10}$c.c. of $\frac{N}{10}$ caustic soda solution represents 1° acidity, and the degrees multiplied by 0.009 will give the acidity in percentage of lactic acid.

If greater accuracy is required 50 c.c. may be taken, and each ½ c.c. will then represent 1° acidity.

The Volumetric Estimation of Fat.—Gerber's method of fat estimation consists in reading the volume of fat brought into the graduated neck of a bottle by

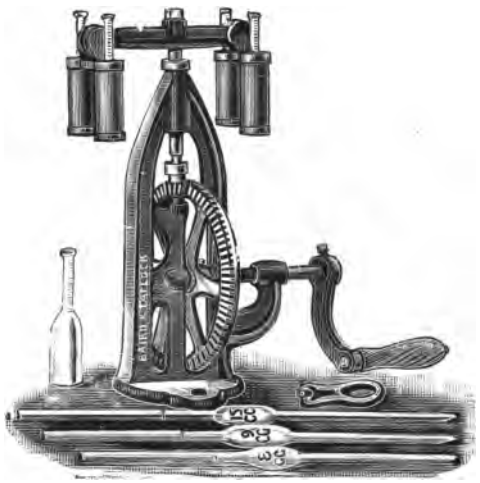


FIG. 13.—Leffmann and Beam Machine.

centrifuging, after dissolving everything in the milk but fat by strong sulphuric acid (the essential part of Babcock's method) with the addition of a little amyl alcohol to help the fat to separate (the essential part of Leffmann and Beam's method (Fig. 13)). In chemical principles Gerber's method is nothing but Leffmann and Beam's, and a description of one only differs in details from that of the other; as however the details of Gerber's method render it more generally suitable, it will be described in preference to its predecessor.

Apparatus.—The essential apparatus consists of:

Acidobutyrometers or test-bottles.—About 20 c.c. capacity with a long stem expanded to a conical bulb at the top, and graduated in percentages of fat; the bottom is

open and contracted to a neck, which is closed by an india-rubber cork when in use. A suitable stand for these is provided.

The test-bottles can now be checked at the National Physical Laboratory, who certify the correctness of the graduated scale.

Measuring Apparatus.—11c.c. pipettes for measuring the volume of milk taken for the test; 10c.c pipettes for measuring the acid; and 1c.c. pipettes for the amyl alcohol. Burettes or automatic measuring apparatus may be used.

Centrifuges.—A centrifuge is necessary to rapidly bring the fat into the graduated neck (Fig. 14A). In the smallest size this consists of two long cups hinged on a bar, which

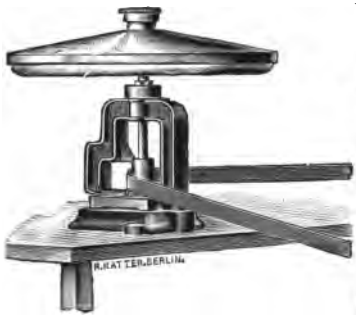


FIG. 14A.

FIG. 14B.

Gerber Machine.

is fixed to the top of a vertical spindle running in plain bearings. This is driven by means of a strap round a loose pulley, or in the Lister machine by means of a string. The larger machines have a disc carrying four or more cups, provided with a cover, and fixed to the top of a vertical spindle running on ball bearings. The driving is performed either as in the two-bottle machine, by a string wound round the spindle, or by a handle. Very large machines are fitted with a steam or water turbine, or an electro-motor (Fig. 14B), and may be provided with heating apparatus to keep the disc warm while running.

Hot-water Tank.—If the disc is not kept warm while running the bottles must be placed in water between 60° and 70° C. before reading, in a small tank.

The Process.—Place a sufficient number of bottles in the stand, open end upwards, and to each add 10c.c of sulphuric acid (see Appendix); add 11c.c. of milk to each by means of the 11c.c. pipette; then add 1c.c. of amyl alcohol (see Appendix).

To measure with a pipette: place the constricted end in the liquid, and draw with the mouth till the level of the liquid is at least one inch above the mark on the upper stem; rapidly remove the mouth, and place the forefinger over the top; the finger must not be wet, though it may with advantage be slightly damp; if this is done with sufficient rapidity, the liquid is above the mark on the upper stem, if it is not it must be drawn up again. Carefully and slightly raise the finger tc allow the liquid to run down slowly to the mark, then stop the flow by pressing the finger on the top; this operation requires some practice, but presents no real difficulty. Keeping the finger pressed down on the top, lift the pipette out of the liquid, and, taking care not to let any drops fall while moving, place the end of the pipette inside the neck of the bottle (Fig. 15); hold the pipette slanting, and let the point touch the side of the neck. On lifting up the forefinger the contents will run out down the side of the bottle. Let the pipette drain a few seconds, and remove it, but do not blow out the last drops. It is essential that the milk be measured with great accuracy, but there is less need for exactitude with the acid or amyl alcohol, as slight variations of these do not affect the results; it is as well, however, to cultivate a habit of accuracy.

After measuring the three liquids, which should float in three distinct layers, with little or no browning at the junction of the acid and milk, insert a cork; the bottle should be held with the left hand, and the cork screwed in, not pushed in, with the right; do not exert too much force or the bottle may break. If there

is marked browning the milk has either been run in too fast or has not been run down the side of the bottle, and the experiment is best repeated.

FIG. 15.—Measuring Milk.

Hold the bottle by the stem, and by the cork, keeping a slight pressure on the cork, and mix the contents by shaking; when all the white particles of curd have disappeared, invert the bottle to allow the acid to run out of the neck, and then turn it upright; repeat this several times till the acid that was in the neck has

completely mixed. The bottle is now almost too hot to hold, from the heat developed by the action of the acid, and its colour is brown; place it in the centrifuge. Treat the other bottles in the same way, and arrange them in order in the centrifuge. It is well to mark each cup with a number to avoid errors.

The shaking of all the bottles may be done at once by means of the author's stand (Fig. 16), in which the bottles are held by being pushed into slits in the india-rubber plate; the hand should be placed over all the corks to prevent them from coming out, and the contents of the bottles from being spilt.

If a number of samples insufficient to fill the disc is being tested, care should be taken to place them symmetrically so as to preserve the balance of the machine; a bottle filled with a mixture of equal parts of acid and water may be kept to make up an even number of bottles should an odd number of samples be tested.

Screw on the cover, and rotate the machine at about 1000 revolutions per minute for three or four minutes; if there is a plain spindle, with a projecting piece, place the eye of the cord on the projection and turn the machine round counter-clock-wise till the cord is wound round the spindle; take the handle in the hand, pull hard, and the disc will spin; usually this must be done twice. If there is a strap, place this half round the pulley, and pull with the right hand in a somewhat downward direction, keeping the strap taut with the left hand, and continue giving sharp pulls till the disc spins rapidly; the pull must be downward as well as forward, or the pulley will not engage the spindle; when the speed slackens, a few further pulls are necessary. If a string with two handles is provided, this is wound once completely round the spindle (or the pulley on the spindle); take a handle in each hand, and pull with the right hand, keeping the string taut with the left hand, and at the end of the pull continue the motion of the left hand to loosen the string round

the spindle; pull back with the left hand keeping the string very loose, and then repeat the stroke. This method of driving requires care, but is, when learnt, a most satisfactory way of spinning the disc; when a sufficient speed is attained the string is allowed to hang loosely, the handles being placed on the bench at each side. If the string is not properly loosened at the end of the stroke, or on the return stroke, it winds up, and the handles must then be immediately dropped, or the hands may receive a nasty blow. Unbleached blind cord is a suitable material for the string, and a good supply should be kept, as the string wears.

During the running the disc may be kept warm by means of a Bunsen burner or a spirit lamp placed underneath near the edge, with a flame so adjusted that it just touches the disc.

The disc should be stopped gently, not suddenly, and the cover unscrewed. Do not take hold of the boss, in the centre of the cover, when the machine is running at high speed.

If the disc is not kept warm, place the bottles, after stopping the machine and removing the cover, in water kept at 60° to 70° C.; the small tank provided with the apparatus is nearly filled with water at the required temperature, and a spirit-lamp or Bunsen burner used to keep it warm.

After one minute the bottles may be read; hold the bottle (Fig. 16) by the top with the left hand and by the cork with the right, at a level with the eye; the position of the bottle should be as nearly vertical as possible. By gently moving the cork, the lower level of the fat column is adjusted to one of the longer lines indicating percentages of fat, and the point on the scale where the lowest part of the curved upper limit stands, is read off; a convenient method of reading is to count first the number of whole percentages of fat (indicated by the longer lines), and then the number of small lines above this, each small line indicating 0.1

per cent. of fat. Fig. 17 shows a sample reading 3.6 per cent. of fat; it is quite easy to read to half a small division or 0.05 per cent.

FIG. 16.—Reading bottles.

Every bottle should be read twice to make sure that there is no error in the first reading.

The following are sources of error:

(*a*) Faulty graduation of the bottle; this is rare.

(*b*) Chemicals not equal to specification; it is important that the specification for these be strictly adhered to.

(*c*) Insufficient mixing of the milk, acid, and amyl

alcohol; this is indicated by the fat being cloudy, and obstinately refusing to become clear.

(*d*) Mixing of the milk and acid before addition of the amyl alcohol; a brown colour of the fat is usually found here.

(*e*) Allowing a portion of the fat to remain in the little conical bulb; if the fat is too low down on the scale for convenient reading, and the cork is not pushed in carefully, the fat is liable to jump up, as it is raised; if the fat is too high, it may partially occupy the conical bulb; in each case the fat must be allowed to run down before reading.

When the corks have been used for some time, they do not fit the necks of the bottles so well as when new, and are liable to slip out, and spill the acid mixture; the times when they are most liable to come out are, when shaking the bottle, when removing it from the disc, when removing it from the warm water, and when pulling down the cork to adjust the fat layer. As acid is detrimental to the clothes, always keep a bottle of ammonia handy, and soak the acid-bespattered garment liberally with this, should any be spilt; if acid is spilt on the hands or face, a copious stream of cold water is the remedy, but do not use ammonia; even strong sulphuric acid spilt on the flesh rarely does harm if washed off at once, and plenty of water is used.

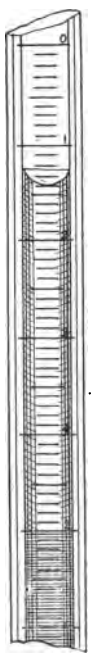


FIG. 17. Gerber Bottle.

When the bottles have been read, turn them cork upwards in the stand, remove the corks, and empty the contents of the bottle into a convenient pot, not down a sink. The pot when full may be emptied down a well-flushed drain; rinse the bottles two or three times in hot water and leave to drain open end downwards; an occasional clean with a brush when the inside becomes dingy is neces-

sary. The corks should be put in a basin and washed several times with hot water and allowed to dry.

Care of the Machine.—The bearings require lubrication; an oil-can and a supply of suitable oil is provided with the machine. If the ball-bearings wear loose, they can be adjusted by means of a collar in the top bearing. If a bottle breaks in the machine remove the cup and wash it carefully, and should acid be spilt on the disc, wash this too.

The machine should be clamped and screwed to a firm bench or table, preferably close to a leg.

If separated milk is being examined a special bottle with narrow neck should be used, and the time of centrifuging increased.

Cream cannot be tested direct, as the scale does not go beyond 9 per cent.; if a thin cream (containing less than 32 per cent. fat) is to be examined, use a 3c.c. pipette in place of the 11c.c. pipette; fill the bottle with acid as before, and then add 8.2c.c. of water from a pipette graduated in $\frac{1}{10}$c.c.; fill the 3c.c. pipette with cream, adjusting the upper level very accurately to the mark, and wipe the outside of the stem; hold the pipette vertically over the centre of the opening and allow the cream to run directly into the water and blow out the last drops; finally, add the amyl alcohol, and proceed as described for milk.

For thicker creams, a small balance weighing to 0.05 gramme (Fig. 18), and a pair or pairs of accurately balanced tin pots are necessary; place sufficient cream in one pot to fill it nearly half full, and put this on one pan, of the balance; place the other pot on the other pan and pour in separated milk or water till exactly balanced. Mix the contents of the two pots, by pouring backwards and forwards several times, and measure out the mixture as directed for thin cream.

Use the table below for the calculation of results; the first column is the reading observed; if thin cream was tested, its percentage of fat is found in the

column headed "Undiluted"; if thick cream was examined, the column headed "Diluted" will give the percentage of fat.

Gravimetric Estimation of Fat.—Of the numerous methods for the estimation of fat, the Adams, the

FIG. 18.—Weighing Cream.

Storch, the Ritthausen, the Werner-Schmid, and the Gottlieb methods all present advantages.

The Adams Method.—Hang up a strip of white demy blotting paper (mill 428), 22 in. long by 2½ in. wide, from which the soluble matter has been extracted by acid alcohol, and pipette 5c.c. of milk over its surface

(Fig. 19), and allow it to air-dry in a place protected from flies; the exact weight of the 5c.c. can be obtained by pipetting another quantity into a basin and weighing it as in the total solids estimation (*q.v.*);

TABLE III.

FOR CALCULATING FAT IN CREAM.

The table should be checked by a gravimetric method, and may require a slight correction added or subtracted which may vary with each pipette.

Reading.	Diluted.	Undiluted.	Reading.	Diluted.	Undiluted.
8.5	63.5	31.8	6.7	49.4	24.8
8.4	62.7	31.4	6.6	48.7	24.4
8.3	61.9	31.0	6.5	47.9	24.0
8.2	61.1	30.6	6.4	47.2	23.7
8.1	60.3	30.2	6.3	46.4	23.3
8.0	59.5	29.8	6.2	45.7	22.9
7.9	58.8	29.5	6.1	44.9	22.5
7.8	58.0	29.1	6.0	44.1	22.1
7.7	57.2	28.7	5.9	43.4	21.8
7.6	56.4	28.3	5.8	42.6	21.4
7.5	55.6	27.9	5.7	41.8	21.0
7.4	54.8	27.5	5.6	41.1	20.6
7.3	54.0	27.1	5.5	40.3	20.2
7.2	53.3	26.7	5.4	39.5	19.8
7.1	52.5	26.3	5.3	38.8	19.5
7.0	51.7	25.9	5 2	38.0	19.1
6.9	50.9	25.5	5.1	37.2	18.7
6.8	50.2	25.2	5.0	36.4	18.3

coil up the strip and dry in the air-bath for fifteen minutes, place in a Soxhlet extractor (Fig. 20), and extract the fat with ether.

The Storch Method.—Place three or four grammes of ignited kieselguhr in a basin, and pipette 10c.c. of milk in such a manner that it is all absorbed by the kieselguhr; dry on the water-bath with occasional stirring to break up lumps, grind fine, and transfer to a fat-free thimble, rinsing out the basin with fresh quantities of kieselguhr; place the thimble in a Soxhlet extractor, rinse the basin, pestle, &c., with ether,

and pour this into the thimble; extract the kieselguhr with ether.

The Ritthausen Method.—See Estimation of Proteids, p. 34.

The extraction in these methods is performed by

FIG. 19.—Putting Milk on Coil.

attaching a weighed flask to the bottom of the extractor to receive the ether containing the fat, and connecting the extractor to an upright condenser; the flask is immersed in water kept warm by a small flame, and the ether continually distils up, is condensed, and runs back into the extractor, from which it siphons back into the flask when the extractor is full.

The Adams coil and the kieselguhr should be extracted about four hours, and casein about one and a half hours, after which the ether is distilled from the flask, and the fat freed from ether by placing in a water oven

FIG. 20.—Soxhlet Extractor.

for twenty minutes, blowing into and rotating the flask every five minutes. After cooling for a quarter of an hour, the flask is weighed, and the increase of weight represents the fat.

The Werner-Schmid Method.—Pipette 10c.c. of milk into a Stokes tube (Fig. 21), add 10c.c. of strong

hydrochloric acid, and heat over a flame *with constant shaking*, till the fat, on standing a short time, collects in a clear layer on the surface; cool the contents of the tube, and add 30c.c. of ether, cork the tube, and shake well; allow the tube to stand till the ether has separated in a clear layer; remove as much ether as possible, preferably by means of washbottle tubes, to a weighed flask; add about 20c.c. more ether, shake well, allow the ether to settle clear, and remove as before, and again add about 20c.c. of ether, shake, allow to settle, and remove the clear layer. Distil off the ether, dry, and weigh as above.

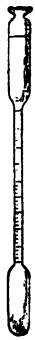


FIG. 21.—Stokes Tube.

Gottlieb's Method.—Place about 10 grammes of milk in a 100c.c. burette, add successively 1c.c. of ammonia, 10c.c. of alcohol, 25c.c. of ether, and 25c.c. of petroleum ether; mix after each addition, and allow to stand; when a clear layer has separated, mix again, allow to separate, mix once more, and allow the clear layer to completely separate. Measure the volume of the upper layer, and evaporate an aliquot portion in a weighed flask, and weigh. The weight multiplied by the total volume and by 100, and divided by the volume taken for evaporation, and the weight of milk, will give the percentage of fat.

Estimation of Milk Sugar.—This may be estimated either polarimetrically, or gravimetrically; the results in either case are expressed as anhydrous sugar.

Polarimetric Estimation.—50c.c. of milk are measured into a dry flask, and a quantity of water equal in cubic centimetres to the sum of

(*a*) The degrees of gravity divided by 20.
(*b*) The percentage of fat divided by 1.8.
(*c*) A quantity to convert scale readings into percentages of anhydrous sugar; if the scale is in angular degrees and a 200 mm. tube is used,

this is 5.43c.c. (or 5c.c. with a 198.4mm. tube).

1.5c.c. of Wiley's acid mercuric nitrate solution (see Appendix) is added, and the whole well mixed by violent shaking. The solution is poured on a dry filter, and a polarimeter tube filled with the clear filtrate.

As an example: the milk has a sp. gr. of 1.032, the degrees of gravity are 32.0, and (*a*) is $\frac{32.0}{20}$ = 1.60c.c.; the fat is 3.60 and (*b*) is $\frac{3.6}{1.8}$ = 2.00c.c.; if an instrument graduated in angular degrees and a 200mm. tube are used (*c*) is 5.43. The water added is 1.60 + 2.00 + 5.43 = 9.03c.c.

The reading is made by placing the tube in the instrument (Fig. 22), focusing the eye-piece on the half-shadow plate when the analyser is so turned that one side is darker than the other, and adjusting the analyser till both sides are equal in intensity; the scale is then read by means of the vernier provided. Several readings should be made, the adjustment to equality being made from either side alternately, and the mean of the readings taken as the correct reading. A blank estimation, *i.e.*, one with a tube filled with distilled water, should always be made, and the reading if to the right subtracted from, or if to the left added to, the reading of the sample. The corrected reading of the scale gives the percentage by weight of anhydrous milk sugar.

Gravimetric Estimation.—About ten grammes of milk are placed in a 100c.c. flask with 60 to 70c.c. water, 5c.c. of Fehling's copper sulphate solution added (see Appendix), and the solution neutralised with caustic soda; the liquid is made up to 100c.c., and the contents of the flask, after mixing, filtered through a dry filter; 50c.c. of the filtrate are placed in a beaker, and a mixture of 30c.c. each of Fehling's copper sulphate and alkaline tartrate solutions (see Appendix) added; the beaker is heated by a flame of such size that the liquid

boils in about four minutes, and it is kept boiling for exactly six minutes. A tube of hard glass, about 1c.m. in internal diameter and 10c.m. long, with one end

FIG. 22.—Polariscope.

drawn out, and plugged with a fairly tight wad of asbestos, is ignited, cooled, and weighed; the drawn out end is inserted in a hole in the cork of a vessel from which the air can be exhausted (*e.g.*, by a filter pump), a small funnel is placed in the wider end, and the liquid carefully poured off from the red precipitate of cuprous oxide; the precipitate is washed several times by decantation with hot, well boiled water, and is then washed into the tube, and the last traces removed

from the sides of the beaker by rubbing with a policeman, and transferred to the tube; the precipitate in the tube is well washed with hot water, and finally with alcohol, and dried. The narrow end of the tube is

FIG. 23.—Washing Precipitate.

connected with an apparatus from which hydrogen can be evolved, and after the stream has passed for a few minutes the part of the tube containing the precipitate is gently heated by a small flame, till the cuprous oxide is reduced to copper; the hydrogen is passed till the tube is cool, and it is then disconnected and weighed; the increase in weight gives the quantity of copper,

from which the quantity of milk sugar can be found by the table below; this multiplied by 200, and divided by the weight of milk taken, gives the percentage of milk sugar.

TABLE IV.

FOR CALCULATING WEIGHT OF MILK SUGAR FROM COPPER REDUCED.

(All weights are in milligrammes.)

Copper.	Milk Sugar.	Copper.	Milk Sugar.	Copper.	Milk Sugar.
120	86.4	215	158.2	310	232.2
125	90.1	220	161.9	315	236.1
130	93.8	225	165.7	320	240.0
135	97.6	230	169.4	325	243.9
140	101.3	235	173.1	330	247.7
145	105.1	240	176.9	335	251.6
150	108.8	245	180.8	340	255.7
155	112.6	250	184.8	345	259.8
160	116.4	255	188.7	350	263.9
165	120.2	260	192.5	355	268.0
170	123.9	265	196.4	360	272.1
175	127.8	270	200.3	365	276.2
180	131.6	275	204.3	370	280.5
185	135.4	280	208.3	375	284.8
190	139.3	285	212.3	380	289.1
195	143.1	290	216.3	385	293.4
200	146.9	295	220.3	390	297.7
205	150.7	300	224.4	395	302.0
210	154.5	305	228.3	400	306.3

Estimation of Proteids. — These may be estimated together by Ritthausen's method, or the casein and albumin may be separated; indirect estimations may be made from the nitrogen by Kjeldahl's method, or from the organic phosphorus and sulphur.

The Ritthausen Method.—Pipette 10c.c. of milk into a beaker, and add 100c.c. of hot water; add 5c.c. of Fehling's copper sulphate solution (see Appendix), and neutralise with caustic soda solution; collect the precipitate either in a weighed Gooch crucible, and on

tared filter paper; remove the precipitate completely from the beaker by means of a policeman, and wash well (Fig. 23). Dry in the water oven, and extract the fat with ether, preferably in a Soxhlet extractor, and dry again till the weight is constant. Ignite the precipitate in the Gooch crucible, or place the filter containing the precipitate in a weighed basin and ignite it (if tared filters are used the tare should also be ignited in a weighed basin); subtract the weight of the ash (corrected if necessary for the ash of the tare) from the weight of the precipitate, and the difference will give the weight of the proteids.

Estimation of Casein and Albumin.—Pipette 10c.c. of milk into a beaker, add 90c.c. of water at 42°–43° C. and 1.5c.c. of a 10 per cent. solution of acetic acid; stir well, and collect and weigh the precipitate as above. [*Note.*—This estimation or that given above may be combined with a fat estimation.] The precipitate is in this case casein.

Raise the filtrate to boiling, and keep for 15 minutes on the water-bath; collect the precipitate as above, and weigh after drying; as fat and ash are absent the extraction and ignition may be omitted. The precipitate consists of albumin.

Estimation of Nitrogen by Kjeldahl's Method.—5c.c. of milk are pipetted into a long-necked hard glass flask; 20c.c. pure sulphuric acid and a drop of mercury are added; a long stemmed bulb is placed in the neck; the flask is supported in an inclined position, and it is heated by a small flame; at first water is driven off, next a considerable amount of frothing takes place, and when this has subsided the flame may be turned up to such a height that the sulphuric acid distils up to and is condensed in the neck; 10 grammes of acid potassium sulphate may be added after the frothing has subsided, but this addition is hardly worth while with milk, as the operation is fast enough without it; the heating is continued till the liquid in the flask is quite colourless.

After cooling, the acid liquid is diluted with about 100c.c. of water, and poured into a flask of at least 1000c.c. capacity (preferably of copper (Fig. 24)), and the hard

FIG. 24.—Kjeldahl Apparatus.

glass flask rinsed out with further quantities of water; the large flask is furnished with a cork carrying a tube which is connected to a condenser, and a tube allowing additions to be made to the liquid. 25c.c. of $\frac{N}{10}$ acid (see Appendix) are accurately measured into a flask, which is placed so that the end of the condenser dips

into the liquid. Through the tube in the cork 100c.c. of a solution of caustic soda containing 300 grammes per litre are poured in, followed by 10c.c. of a 10 per cent. solution of sodium sulphide; the tube is closed, and the contents of the flask mixed by shaking; a flame is placed under the flask, and the ammonia formed by the action of the sulphuric acid on the proteids and liberated by the alkali, distilled over; the distillation takes about half an hour, a volume of approximately 200c.c. being collected. A few drops of cochineal are added to the distillate, and the excess of acid titrated with $\frac{N}{10}$ alkali (see Appendix). A blank experiment, *i.e.*, one without the milk should be performed, and the difference between the volume of alkali used in the blank and the experiment in cubic centimetres multiplied by 0.0014 will give the weight of nitrogen; this multiplied by 100 and divided by the weight of the 5c.c. taken will give the percentage of nitrogen; the nitrogen multiplied by 6.38 may be taken as proteids in the milk.

Estimation of Casein and Albumin Nitrogen.—To 10c.c. of milk add 20c.c. of a saturated solution of magnesium sulphate, and crystals of the salt so long as they are dissolved; allow to stand for some time, and filter off the precipitated casein, and wash this several times with the saturated solution of magnesium sulphate (this is a slow process); place the filter and precipitate in a long-necked, hard glass flask, add 30c.c. of pure sulphuric acid and a drop of mercury, and proceed as above; 150c.c. of soda solution must be added before distillation, and the distillate should be collected in 35 or 40c.c. of $\frac{N}{10}$ acid.

The filtrate is diluted, and the albumin precipitated by tannin; the precipitate is collected on a filter, and the nitrogen estimated as above.

The casein nitrogen and albumin nitrogen may be multiplied by 6.38 to give the amounts of casein and

albumin respectively; the sum of the two is usually a little below the total nitrogen.

Indirect Estimation from Organic Phosphorus and Sulphur.—Estimate the phosphoric acid in the ash of the milk (see p. 15), preferably using 25 grammes of milk; evaporate 25c.c. of the filtrate obtained by adding Wiley's mercuric nitrate to milk for the polarimetric estimation of milk sugar (see p. 31), ignite and estimate the phosphoric acid in the ash of this; multiply the weight of P_2O_5 by 4.42, and this will give the percentage of mineral phosphoric acid in the milk; this subtracted from the percentages of total phosphoric acid will give the organic phosphoric acid, and the casein can be calculated from this by multiplying by 50.8.

Take 25 c.c. of milk, add 10c.c. of strong nitric acid and 1 gramme of sodium carbonate, evaporate to dryness, and add 2 or 3c.c. more nitric acid, and again evaporate; ignite to a fairly white ash, and take up the residue with dilute hydrochloric acid; evaporate to dryness, and boil the residue again with dilute hydrochloric acid, and filter. Just raise the filtrate to boiling, and add barium chloride solution drop by drop so long as a precipitate is produced. Allow the solution to stand 24 hours to complete the precipitation, the first 2 hours preferably in the water oven, and collect the precipitate on a small filter, wash well with hot water, and place filter and precipitate still wet in a weighed platinum dish; ignite over a small flame till the paper is burnt, and then more strongly; the weight of the precipitate (corrected for the ash of the filter) multiplied by 13.7 and divided by the weight of milk taken will give the percentage of sulphur in the milk. For each 71 parts of organic P_2O_5, 32 parts of sulphur belong to the casein; the remainder of the sulphur multiplied by 58.5 will give the albumin.

Estimation of Curd by Lindet's Method.—Estimate the fat and specific gravity as previously described; to 100c.c. of milk add 0.01 gramme of

rennet powder, and keep at 42° C. till curdled; cut up the curd and allow it to settle, and strain off the whey through muslin; cool the whey to 15.5° C. and estimate the specific gravity and the fat as before.

Add the degrees of gravity and the percentage of fat of the milk and subtract the sum of the degrees of gravity and the percentage of fat of the whey; the difference divided by 3·5 will give the percentage of curd available for cheese-making.

In practice the whey obtained during cheese-making may be tested instead of a separate preparation of whey being made; the sample for testing should, however, be removed as early as possible.

The difference in acidity (see p. 16) between the milk and the whey divided by 3.5 will also give a rough estimation of curd.

The Relation between Fat, Solids not Fat, and Specific Gravity.—As the solids not fat of milk are heavier than water, and the fat is lighter, and as, moreover, the extent to which each of these is heavier or lighter respectively is practically constant, it is evident that by means of an appropriate formula any one of the three can be approximately calculated from the other two.

A simple formula which gives a very good approximation is:

$$S = \frac{G}{4} + \frac{F}{5} + 0.14 \quad . \quad . \quad . \quad (1)$$

where S = Solids not Fat per cent. by weight;
G = degrees of gravity; and
F = Fat per cent. by weight.

If it is desired to calculate directly the Total Solids (T) which consists of the sum of the fat and solids not fat we may write

$$T = S + F = \frac{G}{4} + 1.2\,F + 0.14 \quad . \quad (2)$$

As the solids not fat are not generally estimated directly, but are obtained by subtracting the fat from the total solids, the second formula is more useful when the

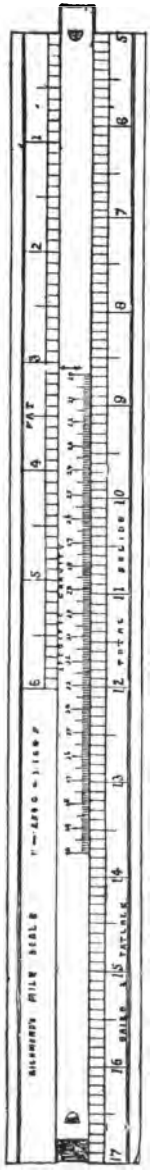


FIG. 25.—Milk Scale.

fat is to be calculated from the specific gravity and total solids, and it may be usefully converted to the form

$$F = 0.833\ T - 0.208\ G - 0.17 \quad (3)$$

or perhaps more simply

$$1.2\ F = T - \frac{G}{4} - 0.14 \quad . \quad . \quad (4)$$

Tables for the easy calculation of solids not fat from fat and specific gravity, and fat from total solids and specific gravity are given at the end of the book. As the method is only approximate the figures are calculated only to the nearest 0.05 per cent. To use these tables find in the upmost horizontal column the specific gravity, and in the vertical column the fat or the total solids; in the space where the columns intersect the figure required is found. If the last figure in the specific gravity is 9, 0, or 1, or 4, 5, or 6, use the figure in the column corresponding to the nearest 0, or 5; if it is 2, or 7, use the column corresponding to 0, or 5, but add in Table X. 0.05, and subtract 0.05 in Table XI.; if it is 3, or 8, use the column corresponding to 5, or 0, and subtract 0.05 in Table X. and add 0.05 in Table XI. Read directions for use of each Table.

The calculation may also be made with the milk scale, which consists of a slide rule (Figs. 25, 26); on one side is marked total solids (1 inch = 1 per cent.), on the other the fat (1.2 inches = 1 per cent.), on the slide is marked specific gravity ($\frac{1}{4}$ inch = 1 degree), and an arrow is placed 0.14 inch from

the end of the scale. If the arrow is placed against the fat, the specific gravity lies against the total solids; *vice versâ*, if the specific gravity is placed against the total solids the arrow will point to the fat.

FIG. 26—Using Milk Scale.

On another part of the rule is a scale for correcting the specific gravity taken at any temperature to 60° F. This is not shown in Fig. 25. On the rule is a scale of degrees of gravity (marked lactometer degrees), and on the slide a scale of temperature; an arrow is placed at 60° F, and this is placed against the degrees of gravity found; the temperature then corresponds to the specific gravity corrected to 60° F; the readings from the milk scale only deviate from those given by the Table within the limits of reading a lactometer.

Though the calculation of the solids not fat or fat can never be so exact as the direct estimation, it is sufficient for many purposes, where small deviations do not affect the conclusions drawn from the results; this method also provides a useful check on the analyses where all three estimations are made, and it is hardly ever found that the deviations of the calculated figures from those estimated exceed 0.2 per cent.

CHAPTER III.

THE ANALYSIS OF LIQUID MILK-PRODUCTS.

THE liquid milk-products are skim-milk, cream, whey, butter-milk, sterilised milk, condensed milk, and sour or fermented milk.

Skim Milk is treated exactly as milk; as the fat globules are very small and few, the estimation of fat requires more care; the period of revolution of the disc in the Gerber method should be increased, and a correction of 0.05 per cent. should be added to the reading unless a special tube is used. The gravimetric methods of Adams, Storch, and Ritthausen tend to give slightly low results, and those of Werner-Schmid or Gottlieb are preferable. In the estimation of the proteids by Ritthausen's method, the extraction of the fat may be omitted, and the percentage of fat found subtracted from the results.

Cream requires several modifications. The *specific gravity*, except of a thin cream, is difficult to estimate, and this is usually omitted. It is advantageous to add an equal volume of alcohol to the cream before drying for the *total solid estimation*, as there is then no skin to be broken. The *fat* may be estimated by macerating the total solids with ether or amyl alcohol, carefully decanting and repeating the maceration, &c., about half a dozen times; the solids not fat are weighed directly, and the fat found by difference. The *ash estimation* is made on the solids not fat. The Werner-Schmid method is, however, excellent for cream, with the modi-

fication that 2 or 3 grammes should be weighed, into the Stokes tube, and the weight made up to 10 grammes with water; the method is then carried out as for milk.

The fat may be calculated from the total solids with a close approach to accuracy by the formula

$$F = 1.104\,T - 10.4$$

or by the following table:

TABLE V.

FOR CALCULATING PERCENTAGE OF FAT IN CREAM FROM TOTAL SOLIDS.

Total Solids.	Fat.	Solids not Fat.	Total Solids.	Fat.	Solids not Fat.
60	55.8	4.2	44	38.2	5.8
59	54.7	4.3	43	37.1	5.9
58	53.6	4.4	42	36.0	6.0
57	52.5	4.5	41	34.9	6.1
56	51.4	4.6	40	33.8	6.2
55	50.3	4.7	39	32.7	6.3
54	49.2	4.8	38	31.6	6.4
53	48.1	4.9	37	30.4	6.6
52	47.0	5.0	36	29.3	6.7
51	45.9	5.1	35	28.2	6.8
50	44.8	5.2	34	27.1	6.9
49	43.7	5.3	33	26.0	7.0
48	42.6	5.4	32	24.9	7.1
47	41.5	5.5	31	23.8	7.2
46	40.4	5.6	30	22.7	7.3
45	39.3	5.7	29	21.6	7.4

This method is not available in the case of clotted or Devonshire cream.

The **milk sugar estimation** by the polariscope requires modification as the cream must be more highly diluted; it is best to weigh out 50 grammes of cream, make up to 100c.c., and add 1c.c. of Wiley's acid mercuric nitrate, and polarise.

The milk sugar is found by the formula

$$\text{Milk sugar} = R \times \frac{1}{1.05} \times 0.95 \times \frac{100 - 1.076\,F \times W}{W}$$

Where R = Reading in angular degrees with a 200 mm. tube.
F = percentage of fat by weight.
W = weight of cream taken.

The gravimetric estimation will require slight correction for the volume of the fat, and the formula

$$\text{Milk sugar} = 2M \times \frac{100 - 1.076\,F \times W}{W} \text{ may be used.}$$

M = weight of milk sugar obtained from the table on p. 34.

The **proteid estimation** is carried out as for milk; it saves time, however, to dry the cream and to extract the bulk of the fat before submitting the sample to Kjeldahl's method, as fat is attacked but slowly by sulphuric acid and mercury.

Butter-milk is analysed in exactly the same way as milk.

Whey is treated as milk; it contains, however, no casein, but gives a small precipitate consisting of albumoses, which by the methods given would be estimated as casein.

Sterilised milk can be analysed by the methods given for milk; the polarimetric estimation of milk sugar tends to be low, owing to change in the milk sugar on heating, and the gravimetric method should be used. The albumin behaves like casein, as it is rendered insoluble in dilute acetic acid and magnesium sulphate solutions; the estimation of casein and albumin can, however, be made by the indirect method from organic phosphorus and sulphur. The total nitrogen is unaffected. Ritthausen's method should not be used for the estimation of fat in sterilised or condensed milk.

Condensed milk, if unsweetened, may be analysed by diluting one part by weight with two parts of water and boiling, and treating in the same way as sterilised milk; the results must, of course, be multiplied by 3.

Sweetened condensed milk should be similarly diluted and analysed; it is not generally heated to an

extent sufficient to affect the milk sugar, and both gravimetric and polarimetric methods of estimating sugar must be employed; the gravimetric method gives only the milk sugar, and the polarimetric method indicates both sugars; the latter, of course, gives a much higher result; the difference between the two results multiplied by 0.831 will give the cane sugar.

The estimation of the sugars is not very exact.

The Werner-Schmid method for the estimation of fat should not be used, and the Adams method is recommended.

Sour milk is difficult to analyse, and the results are generally less satisfactory than those obtained with fresh milk. If approximate results only are wanted, such as would be furnished by a determination of specific gravity and fat alone, the following modification of Weibull's method may be used; measure the sour milk, and to each 100c.c. add 5c.c. of a solution of ammonia (1 part of ammonia, sp. gr. 0.880, to 4 parts water); shake gently, and allow to stand till the precipitated casein is all dissolved; the specific gravity is estimated by a lactometer, and a correction (usually about 2.7 or 2.8 degrees), found experimentally by noting the decrease of specific gravity in fresh milk treated similarly, is added; the fat is estimated by the Gerber method (or one of the gravimetric methods), and the result is increased by one-twentieth; this method is available if the milk is not too old, and serves excellently for control work.

An estimation of total solids may be made after neutralising the acid with caustic soda solution; an estimation of the acidity is made as usual (p. 16), a weighed quantity being used instead of a measured amount, and a proportionate amount of soda solution added to the weighed quantity of milk taken for total solid estimation; the first period of drying should be for only two hours, and the subsequent periods should be strictly one hour; from the weight of total solids

0.0022 gramme should be deducted for each cubic centimetre of $\frac{N}{10}$ soda added.

The ash is estimated as usual, but 0.0053 gramme should be deducted from the weight of the ash for each cubic centimetre of $\frac{N}{10}$ soda solution added.

The fat estimation is preferably made by the Storch method (an addition of soda solution being made as in the total solid estimation) or the Gottlieb method; the Werner-Schmid method is also available, though it tends to give high results with very sour samples owing to the solubility of lactic acid in ether.

Other determinations are made as for milk, except that the quantities taken are all weighed and not measured. The total nitrogen is a useful datum.

The methods used in the Government laboratory include the determination of alcohol, volatile acids, and ammonia, and from these, the solids lost by the various fermentations undergone by the milk are reconstructed. The following description is condensed from Dr. Thorpe's report:

Alcohol.—To 75 grammes of sour milk half the caustic soda solution necessary to neutralise is added, and the mixture is distilled; to the distillate is added 0.5c.c. $\frac{N}{10}$ soda solution, and this mixture again distilled; the final distillate is made up to the original bulk, and the specific gravity estimated. The difference in degrees of gravity between the specific gravity and 1000 multiplied by 0.977 gives the percentage by weight of milk sugar converted into alcohol.

Volatile Acid.—Ten grammes of milk are neutralised to the extent of one half, and a little phenolphthalein added; the mixture is evaporated to dryness on a water-bath with frequent stirring, and after the addition of 20c.c. boiling distilled water, $\frac{N}{10}$ soda solution is added till a pink colour just appears. The difference between the number of c.c. of $\frac{N}{10}$ soda

solution used in this experiment, and that required for the original acidity of 10 grammes of milk, is multiplied by 0.0255 to give the percentage of milk sugar converted into volatile acid.

Ammonia.—Two grammes of milk are diluted to 100c.c. and filtered clear. Ten c.c. of the filtrate made up to 50c.c. with distilled water are compared in tint with a solution of ammonium chloride solution (1c.c. = 0.01 milligramme NH_3) in 50c.c. water containing 10c.c. of a solution of 2 grammes fresh milk acidified in 100c.c. after the addition of 2c.c. of Nessler solution (see Appendix) to each. The number of c.c. of ammonia solution required to produce the same tint multiplied by 0.026 gives the percentage of casein converted into ammonia.

The three amounts are added together, and constitute the total correction for solids lost by fermentation.

CHAPTER IV.

THE APPLICATION OF ANALYSIS TO THE SOLUTION OF PROBLEMS.

The Detection of Adulteration.—The principal forms of adulteration of milk are the addition of water and the removal of cream.

The detection of water is based on the reasoning that while the water natural to milk contains solids not fat, added water is free from these. The amount of solids not fat is nearly though not quite constant, and rarely falls below 8.5 per cent. or rises much above 9.2 per cent.; numerous cases, however, are on record of solids not fat below 8.5.

The removal of cream is detected by a deficiency in the fat; this varies much more than the solids not fat, but comparatively rarely falls below 3.0 per cent.

The probability of samples falling below 8.5 per cent. of solids not fat, and 3.0 per cent. of fat is indicated by the following Table which gives the number of samples per 100,000 which may be expected at each percentage named; it is assumed that each sample represents a churn of milk, *i.e.*, that the milk is the mixed product of several cows.

TABLE VI.

Percentage of Solids not Fat.	Number of Samples.	Percentage of Fat.	Number of Samples.
8.4 to 8.5	1892	2.9 to 3.0	370
8.3 to 8.4	242	2.8 to 2.9	209
8.2 to 8.3	27	2.7 to 2.8	87
8.1 to 8.2	22	2.6 to 2.7	37
8.0 to 8.1	8	2.5 to 2.6	16
Below 8.0	2	Below 2.5	13

By Clause 4 of the Sale of Food and Drugs Act, 1899, the President of the Board of Agriculture is empowered to lay down limits below which a presumption is raised that milk is not genuine, and he has fixed 8.5 per cent. of solids not fat and 3.0 per cent. of fat. The effect of this is that the onus of proving that milk taken under the Sale of Food and Drugs Acts falling below these limits is genuine, lies on the vendor, and for most practical purposes milk below these limits is taken as adulterated.

It is generally, though not invariably, found that, in milk falling below 8.5 per cent. of solids not fat, the deficiency lies chiefly on the milk sugar, and that the proteids and ash are normal; a percentage of total nitrogen above 0.5 and a percentage of ash above 0.7 in a milk below 8.5 per cent. of solids not fat will afford strong evidence that the milk is genuine, while figures for total nitrogen and ash low proportionately to the solids not fat will strengthen the conclusion that the milk is watered.

There appears to be no chemical means of distinguishing between fat naturally low and fat lowered by the abstraction of cream; the most numerous instances of fat below 3.0 per cent. naturally occurring have been found in April, May, June, and July, and they are especially rare in October, November, and December.

The percentage of added water may be calculated by the formula:

$$\text{Added water} = \frac{8.5 - S}{8.5} \times 100. \quad (S = \text{solids not fat.})$$

A formula which gives a nearer approach to the probable amount is:

$$\text{Added water} = \frac{36 - (G + F)}{36} \times 100.$$

$$(G = \text{degrees of gravity.} \quad F = \text{fat.})$$

The amount of cream abstracted may be calculated by the formula:

$$\text{Cream abstracted} = \frac{3 - F}{3} \times 100. \quad (F = \text{fat.})$$

This gives the minimum percentage of cream abstracted, and the more probable amount is obtained by substituting the average percentage for the month as given in chap. i. (p. 3) for 3 in both places.

Detection of Preservatives. *Boric Acid.*—To a little milk add a few drops of phenolphthalein, and caustic soda solution drop by drop till a faint pink colour is produced; place some of the milk in two test-tubes, dilute one with an equal volume of water, and the other with a *neutral* 50 per cent. solution of glycerine; in the absence of boric acid the two tubes will have almost the same colour, in its presence the glycerine tube will be the lighter, and usually white.

As an alternative method the milk or its ash may be made distinctly, but not strongly acid with hydrochloric acid, and a piece of turmeric paper dipped into the solution; on drying, the paper turns pink in the presence of boric acid, and is turned a greenish-black by alkalies.

Formaldehyde.—Dilute a little milk with an equal bulk of water in a test-tube; pour carefully down the side of the tube a little 90 per cent. commercial sulphuric acid; a bluish colour is developed at the junction of the acid and milk in the presence of formaldehyde. This blue colour may also be observed during the estimation of fat by the Gerber process (p. 17).

Salicylic Acid.—This acid gives a violet colour with ferric chloride, and is best tested for by extracting the filtrate produced by treating milk with acid mercuric nitrate (p. 31) with ether, evaporating the ether, taking up the residue with a little water, and adding a little ferric chloride solution.

Hydrogen Peroxide.—Mix the sample with a little

fresh milk, and add a small amount of para-phenylene-diamine or ortol; a blue or red colour will be developed if hydrogen peroxide be present.

Reaction of Milk with Hydrogen Peroxide.—Fresh milk when treated with a little para-phenylene-diamine or ortol (a photographic developer) and a drop of hydrogen peroxide gives a deep blue (with the diamine) or a brick-red (with ortol) colouration within a few seconds. Milk heated above 80° C. remains white.

The Cause of Poor Milk.—The detection of added water and of a deficiency of cream would be an obvious explanation of the cause of milk being poor. If it is normal in composition, but very white, and the fat separated in the Gerber process is nearly free from colour, this would show that the poverty of the milk had been fallaciously inferred from its lack of colour; if this is not the case, the test given above should be applied, and the soluble albumin estimated (p. 35); a deficiency of albumin below 0.35 per cent., or the non-production of colour with para-phenylene-diamine or ortol will show that the milk has been heated, and as the cream rises very slowly on heated milk, the milk has been called poor because cream is not apparent in a short time. Occasionally a sample called poor turns out to contain a high percentage of fat; this would show that the milk has been standing long enough for the cream to separate and it is then divided into a rich and a poor portion.

A not unusual practice is for the servants in a household to pour off a portion of milk from a can which has stood some time for their own consumption, and remove the cream, and to send the rest of the milk, thus impoverished, for the consumption of the other members of the household.

The Cause of Sweet Milk.—Milk is sometimes alleged to be sweet; if the milk sugar estimated polarimetrically is much higher than that determined gravimetrically the presence of cane sugar may be inferred; if all the figures for solids not fat, ash, sugar, and

proteids are equally high, the milk has simply been concentrated, usually by boiling; the solids not fat have

FIG. 27.—High-speed Centrifuge.

been found as high as 17.5 per cent. in a case of this kind.

The Cause of High Colour.—If the colour is yellow and the fat separated by the Gerber process is very much darker than usual, and the cream separating is much yellower than the skim milk, the high colour is natural.

If this is not the case artificial colours should be tested for; annatto is detected by making the milk alkaline with sodium bi-carbonate, immersing a strip of filter paper in it, and allowing to stand till next day; in the presence of annatto the strip is stained brownish. Coal-tar dyes of the azo group give a pink colour when

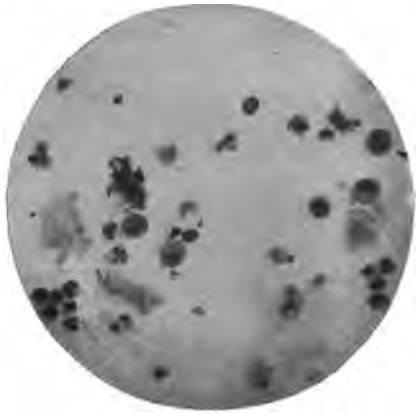


FIG. 28.—Blood in Milk.

a mineral acid is added to milk, and this is usually seen in the Gerber test. Other artificial colours are practically never used.

A pink colour is generally due to blood; to detect this the milk is warmed to 50° C., and separated in a high speed centrifuge (Fig. 27); a bright red deposit at the bottom of the tube may be taken as blood; the microscopic appearance of the corpuscles is given in Fig. 28.

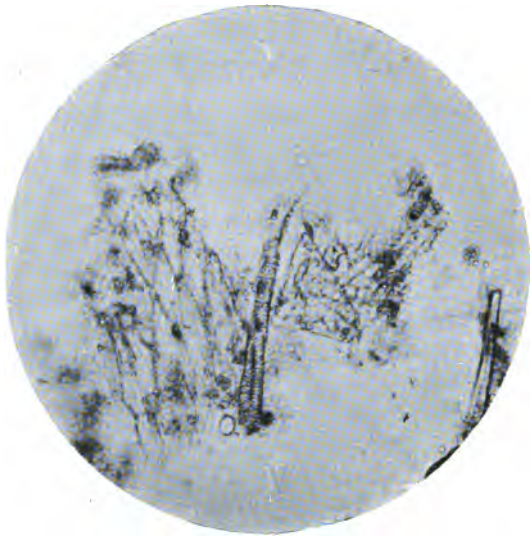
The Cause of Sour Milk.—Practically the only cause of milk turning sour is the formation of lactic acid by the action of micro-organisms. Milk curdles on boiling when the acidity reaches about 33°, and spontaneously in the cold when the acidity is about 80°; milk curdled by boiling generally contains somewhat

hard lumps of curd, and the acidity of the whey may be 25° and upwards; at lower temperatures the curd is softer, and if the acidity is appreciably below 80°, this

FIG. 29.—Using Microscope.

indicates that the milk has been kept warm, but not warm enough to inhibit microbial action.

Custards made with eggs, especially if much sugar is added, inevitably curdle if heated to too high a temperature; the liquids from such samples are usually high in total solids (about 16 per cent.), have a low acidity,



FIGS. 30, 31.—Vegetable Matter in Milk.

and are nearly clear, characteristics which permit of the cause of curdling being established.

Occasionally milk is alleged to be sour because it turns blue litmus red; all milk does this, as well as turning red litmus blue, owing to the amphoteric reaction of the phosphates and citrates of the milk.

The three popular superstitions that milk can be kept warm, *i.e.*, a little above blood heat for several hours without change, that custards can be heated to any temperature, and that blue litmus can be used as a test for sour milk, though all fallacious, are responsible for many allegations of sour milk. The peptonisation of milk with powders which are insufficiently alkaline may cause it to curdle, and to be thought to be sour.

The Cause of Unusual Taste.—Milk on boiling acquires a taste, and the tests for heated milk (p. 52) and soluble albumin (p. 35) will show this. Mixture with dirty water may give an evil taste to milk, and usually the establishment of the presence of water is all that can be done to explain this; if an alkali is added to milk, the taste is soapy and the smell fishy, and an increase in ash and its strongly marked alkalinity will detect the cause. If the taste is due to a fermentation other than the normal lactic one, or to the food of the cattle, it is usually difficult to detect the cause by chemical analysis.

The Cause of Dirty Milk.—Dirty milk almost invariably deposits a sediment on standing or centrifuging; the milk is carefully decanted, the sediment washed with water, and allowed to settle again and examined under the microscope (Fig. 29). Sharply defined vegetable cells (Fig. 30), indicate that the finer particles of the food given to the cattle probably at milking time have fallen in; less well defined vegetable cells (Fig. 31), stained yellowish are probably derived from fæcal matter; small hairs and various fibres (cotton, wool, &c.) show the presence of household dust; transparent irregular particles which do not polarise are quartz, and are due to road dust; this latter also

gives a strong reaction for iron on treating the sediment with hydrochloric acid, diluting, and testing with potassium ferrocyanide which gives a blue colour with iron salts.

Detection of Adulteration of Cream.—As there is no standard for cream, it may contain any percentage of fat, and still be cream; there is a practical standard of "thickness" which the purchaser mentally estimates, and judges the value of the cream thereby. Artificial thickening is sometimes resorted to, and gelatinised starch, gelatine and "viscogen" (a solution of lime in cane-sugar syrup) are added.

Starch is detected by the blue colour produced on adding a solution of iodine in potassium iodide.

Gelatine is found, if present, by diluting the cream with water and adding a little acid mercuric nitrate solution (see Appendix); the filtrate if gelatine is present is usually turbid, and gives a precipitate on the addition of a saturated solution of picric acid.

Viscogen raises the percentage of lime in the ash; the lime on an average amounts to 22 per cent. of the ash, and its ratio to phosphoric acid (CaO to P_2O_5) is 1 : 1.3. Viscogen raises not only the percentage in the ash, but also the ratio; it also renders the ash markedly alkaline. The percentage of sugar polarised as milk sugar will exceed 52.5 per cent. of the solids not fat.

Preservatives are detected in the same way as for milk.

Adulteration of Skim Milk.—The President of the Board of Agriculture has fixed 9.0 per cent. as the limit for total solids in skim milk. Percentages below this are presumed to be caused by the addition of water.

Rennet is sometimes added to skim milk, and even to whole milk, usually with the idea of causing curdling when the milk is warmed. Its presence may be inferred, if the milk curdles on warming to 40° C., and the acidity is less than 25°; the whey on neutralising to

an acidity of 12° will cause fresh milk to curdle at 40°, and the amount of lime in the whey does not exceed 0.06 per cent.

Detection of Foreign Fats in Milk and Cream.—By means of an emulsifying apparatus, foreign fats (margarine fat, cocoa-nut oil) are mixed with separated milk and the product sold as milk or cream. The casein should be precipitated from a considerable amount of milk (p. 35) dried, extracted with ether, and the fat examined as butter fat (pp. 62 *et seq.*).

CHAPTER V.

THE ANALYSIS OF BUTTER.

Estimation of Water.—Weigh a small round basin (Fig. 32) about 3 in. in diameter, containing a small rod; place 5 to 10 grammes of butter therein, and weigh again; heat the basin over a very small flame, or on a sand-bath, and stir *constantly* till frothing has ceased, cool, and weigh again. The loss of weight indicates water, and this multiplied by 100 and divided by the weight of butter taken gives the percentage. The flame should be of such a size that the butter takes at least a minute to become melted.

As an alternative method about 2½ grammes of butter may be weighed in a flat-bottomed basin, just melted in the water-oven, and 1½c.c. strong alcohol mixed with the melted fat; the basin is placed in the water oven for two hours, cooled, and weighed. The loss of weight gives the water.

Estimation of Curd and Salt.—Wash the fat from the basin after driving off the water by nearly filling it with ether or amyl alcohol, and carefully decanting the liquid after the solid portion has settled, and repeating this four times; if amyl alcohol is used, it should be hot; the residue is dried in the water-oven for two hours, and weighed after cooling. This represents curd and salt (if present).

Extract the salt from the curd with hot water, and filter the solution; wash the residue and the filter, and cool the filtrate; add a few drops of potassium chromate

solution, and titrate with $\frac{N}{10}$ silver nitrate solution till a faint red colour just appears; each cubic centimetre used is equal to 0.00585 gramme salt.

Estimation of Casein.—Extract another portion of curd with dilute ammonia till no lumps are left;

FIG. 32. Water Estimation.

filter, and wash the residue; add dilute acetic acid till a white precipitate falls, and collect this in a weighed Gooch crucible or on tared filter papers, as on p. 35. Extraction and ignition may be omitted. The precipitate is casein.

Tests for Preservatives.—Boric acid may be detected by melting the butter at a low temperature, and testing the aqueous portion with turmeric paper as directed on p. 51.

If found, the estimation is carried out by weighing 25 grammes of butter, adding 25c.c. of a solution containing 6 grammes of milk sugar and 4c.c. N acid per 100c.c., melting the butter at a low temperature, and stirring well; the aqueous portion is allowed to settle, and 20c.c. are withdrawn, and placed in a small beaker and boiled; a little phenolphthalein solution is added, and the solution titrated while still boiling with $\frac{N}{2}$ caustic soda solution (see Appendix) till a faint pink colour occurs; the reading of the burette is noted, 12c.c. glycerine are added, and the solution titrated again (boiling is not necessary) till a pink colour appears; the difference between the reading of the burette, and the first reading multiplied by 0.0374, will give the weight of boric acid (as H_3BO_3), and this multiplied by $5 + 0.05$ W (W = percentage of water) will give the percentage of boric acid. Where the percentage of water is not estimated the figure 5.65 may be used for $5 + 0.05$ W, and as an approximate figure the number of cubic centimetres of $\frac{N}{2}$ soda solution used between the first and second titrations may be multiplied by 0.2.

Sulphites are detected by the smell of sulphurous acid given off on acidifying the aqueous portion; formalin will give its characteristic reaction (p. 51) if a little of the aqueous portion is added to milk; fluorides are detected by adding a solution of calcium chloride to the aqueous portion, filtering, igniting the precipitate, taking up with dilute acetic acid, and treating the insoluble portion with strong sulphuric acid in a platinum dish over which is inverted a glass plate coated with beeswax through which one or two lines are scratched; on warming the sulphuric acid, hydrofluoric acid is given off in the presence of fluorides, and this etches the

portions of the glass exposed by scratching through the beeswax, and the marks are visible on melting and wiping off the beeswax.

The Examination of the Fat.—Butter fat is of peculiar composition, consisting of complex glycerides containing lower fatty acids, chiefly caproic and butyric acids; these are characterised by being soluble in water, and volatile with steam, while the fatty acids of almost all other fats are insoluble and non-volatile; furthermore the presence of the lower fatty acids in the glycerides causes them to have a softer consistency than if only the insoluble acids were present, and a comparatively small amount of the acids of the oleic and more unsaturated series is present. To obtain a fat of the consistency of butter without the lower fatty acids, a larger amount of acids of the oleic, &c., series must be present. The presence of the lower fatty acids gives a high specific gravity to the glycerides, and causes them to crystallise badly.

In addition to these facts on which the broad principles of butter analysis are based, certain vegetable oils, especially sesamé oil, give characteristic reactions, and it has been recommended that by international agreement all margarine shall legally be made to contain 10 per cent. of sesamé oil.

The addition of margarine to butter may be detected by—

(*a*) A lowered proportion of volatile acids;
(*b*) A lowered proportion of soluble and increased proportion of insoluble acids;
(*c*) An increased mean molecular weight;
(*d*) A decreased density;
(*e*) A more marked crystallisation;

properties all chiefly depending on the lowering of the amount of caproic and butyric acids in the glycerides, and—

(*f*) An increased iodine absorption;
(*g*) An increased refractive index;

properties chiefly depending on the increase of the

unsaturated acids in the glycerides; to these may be added—

(*h*) A turbidity of the fat on melting at a low temperature, a property which depends on the fact that mixing margarine with butter often causes overworking, which gives rise to turbidity.

Owing to the natural variations of the composition of butter fat, and to a less degree of the composition of adulterants, the detection of small quantities of margarine is difficult, and unless sesamé oil can be found, impossible in minimal amounts; as only three principles underlie all the methods, one of which has little value, a multiplication of tests does not greatly assist.

Preparation of the Fat for Analysis.—Place 20 to 50 grammes of butter in a small beaker, and put this in the water oven till melted; observe the fatty layer, whether clear or turbid; pour as much as possible of the fat into a dry filter, taking care that none of the aqueous portion accompanies it, and filter in the water oven, collecting the clear fat in a small beaker.

Estimation of the Volatile Fatty Acids. *Reichert-Wollny Process.*—Place a flask of 300c.c. capacity on one pan of a balance and tare it; add 4.5 grammes to the weights, and run in the melted fat till the flask is weighed down, and place a further 0.5 gramme weight on the other pan; continue the addition of the fat cautiously, till the weight is exact, if necessary removing a surplus with a small pipette. It is not necessary to wait for the fat in the flask to cool before making the final adjustment, as the error involved in weighing warm fat is within the limits of error of the final titration, nor is it necessary to weigh more accurately than to 0.005 gramme.

Add 10c.c. of alcohol and 2c.c. of a 50 per cent. solution of caustic soda (see Appendix), preferably from a special measuring apparatus (Fig. 33); without delay connect the flask to a T-piece, one end of which is attached to an upright condenser, and the side tube of which is closed, and pointed in an upright direction;

the T-piece should be inclined. Heat on a water-bath for half an hour, and at the expiration of this time turn the T-piece so that the side tube slants downwards, and open this; distil off the alcohol for a quarter of an hour. Add, through the T-piece, 100c.c. of hot water,

FIG. 33. Caustic Soda Apparatus.

which has been previously boiled for at least fifteen minutes, and when all the resulting soap is dissolved, two or three pieces of pipe-clay, and 40c.c. of sulphuric acid solution (see Appendix); remove the flask from the T-piece, and attach by means of a cork to a bulb tube

attached to a condenser, the dimensions of which are given in the sketch (Fig. 34). Support the flask on a piece of asbestos card in which is cut a hole 5c.m. in diameter and heat with a very small flame, till the fatty acids float in a clear layer on the surface of the liquid; then turn up the flame to such a height that 110c.c. distil in from twenty-eight to thirty-two minutes.

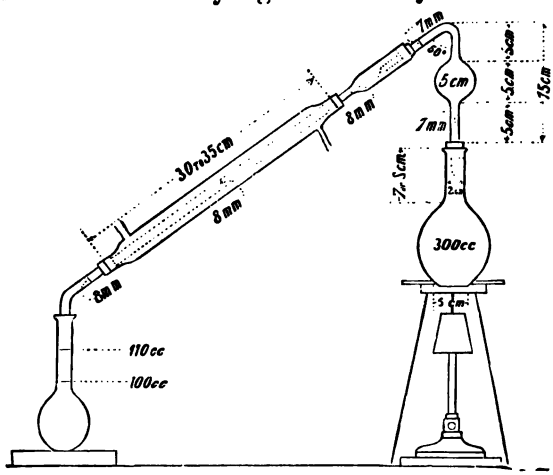


FIG. 34. Reichert-Wollny Apparatus.

Collect 110c.c., mix the distillate, and filter through a dry filter; use the first few c.c. to wash out a 100c.c. flask, and collect exactly 100c.c. of the filtrate, and transfer this to a beaker, add a little phenolphthalein and titrate with $\frac{N}{10}$ baryta, strontia, or soda solution till a pink colour appears; pour this back into the flask, and again into the beaker, and if the colour is discharged continue the titration. From the number of cubic centimetres used, subtract the figure obtained in a blank experiment, and multiply the result by 1.1 to obtain the Reichert-Wollny figure.

As an alternative 10c.c. of glycerol may be substituted for the alcohol, and the saponification effected by

heating over a flame, till the solution is quite clear; a considerable amount of frothing occurs when saponification takes place; the method, due to Leffmann and Beam, is conducted similarly to the Reichert-Wollny method, and the results are practically the same.

Estimation of Soluble and Insoluble Fatty Acids and mean Molecular Weight.—Weigh a glass flask, which has been previously well boiled with caustic alkali solution; add about 4 grammes of butter fat, and weigh again. Run in 50c.c. $\frac{N}{2}$ alcoholic soda solution (see Appendix), attach the flask to an upright condenser, and boil for a quarter of an hour; add a few drops of phenolphthalein solution and titrate with $\frac{N}{2}$ hydrochloric acid till the pink colour is discharged. As the alcoholic soda solution alters in strength it must be checked against the $\frac{N}{2}$ hydrochloric acid, and the value in terms of the acid solution obtained of 50c.c. of alcoholic soda; from this subtract the volume of $\frac{N}{2}$ hydrochloric acid used in titrating after saponification, and multiply by 2.805 and divide by the weight of butter taken; the figure obtained will be the percentage of potash required for saponification.

Wash out the alcoholic solution into a large basin, and evaporate the alcohol on the water-bath; add enough hot water to make the bulk up to 150 to 200 c.c., and then add sufficient $\frac{N}{2}$ hydrochloric acid solution to make with the volume already used in the titration 1c.c. more than the quantity equal to the 50c.c. alcoholic soda added; heat on the water-bath till the insoluble fatty acids float on the surface in a clear layer, and filter through a wet filter; wash out the basin with hot water till the fatty acids are transferred to the filter, and wash them well on the filter, stirring them up with the jet of water; at least a litre of water is required for washing. With a good filter the fatty acids do not run through; when washed, allow all the

water to run out, and wash the filter with hot alcohol, collecting the filtrate in a weighed flask, till all the fatty acids are removed; evaporate the alcohol, and dry in the water-oven till the weight is constant; this will give the insoluble fatty acids.

To estimate the soluble fatty acids, add a little phenolphthalein solution to the filtrate from the insoluble fatty acids, and titrate with alcoholic alkali till a pink colour appears; from the volume used calculate the equivalent of $\frac{N}{2}$ acid, from this subtract the 1c.c. added in excess of the soda added, multiply by 4.4, and divide by the weight of butter taken; this will give the soluble fatty acids in terms of butyric acid.

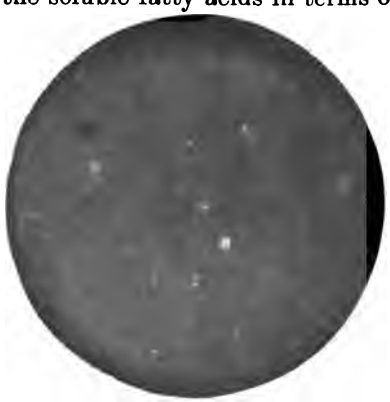


FIG. 35.—Butter.

Estimation of Density.—Follow the directions on p. 10, with the following modifications: weigh the tube full of water at 37.8 °C. (100 F.) instead of at 15.5° C; dry the tube before filling it with the fat, and take the density at 37.8° C. instead of at 15.5° C.

Examination under Polarised Light. — Place a small portion of the butter (not butter fat) on a microscope slide, and press down a cover-glass thereon; examine with a microscope furnished with a polariscope, using a 1 inch or ½ inch power, focus with the Nicols parallel, and then cross them, shielding the slide from light except that which has passed through the polariser. Genuine butter appears nearly uniformly dark, while crystalline fats show a more or less well-marked lighting in portions of the field.

Old butters, especially those which have been

submitted to vibrations, and butters prepared by processes in which the cream is churned soon after heating and cooling may show (Fig. 35) a somewhat crystalline appearance, but generally this is due to margarine (Fig. 36); this test though very rapid may not be reliable.

Estimation of Iodine Absorption.—Weigh about 0.4 to 0.5 gramme of fat in a stoppered bottle, dissolve in 10c.c. of chloroform, and add 20c.c. of v. Hübl's iodine solution (see Appendix). At the same time mix 10c.c. of chloroform with 20c.c. of iodine solution, and place both bottles in a dark place for 4 hours. Add 15c.c. of a 10 per cent. solution of potassium iodide solution to each, and about 200c.c. of water, and titrate with $\frac{N}{2}$ sodium thiosulphate solution (see Appendix) till the colour on shaking is removed from both aqueous and chloroformic solutions; a little starch solution may be added when the colour is very pale, and the titration carried on till all blue has disappeared, but its use is not absolutely necessary.

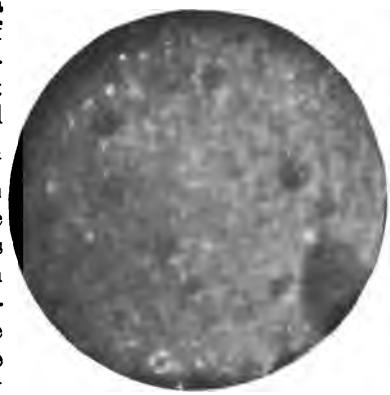


FIG. 36.—Margarine.

The number of c.c. of thiosulphate multiplied by the value of 1c.c. in terms of iodine used in the blank experiment gives the total amount of iodine added to the fat; the number of c.c. of thiosulphate similarly multiplied used in the actual experiment gives the weight of iodine not absorbed by the fat, and the difference between these two gives the quantity absorbed, and this multiplied by 100, and divided by the weight of fat taken is the iodine absorption.

Determination of Refractive Index.—The Zeiss Butyro-refractometer is employed for the determination of the refractive index; it consists of two water-jacketed prisms, between which the substance is

FIG. 37.—Refractometer.

placed, a mirror to reflect light through these, an eye piece with a scale in it by which the refractive index is read off, and a thermometer for observing the temperature. An apparatus for providing a stream of water of constant temperature can be used, or in default of this a stream of water warmed to the required

degree can be run through the jacket by india-rubber tubes from any fair-sized vessel; though it is an advantage to use water at a constant temperature, the cooling of a large bulk of water is sufficiently slow to keep

FIG. 38.—Refractometer.

the prisms at practically a constant temperature during the reading.

To make an observation, take the refractometer (Fig. 37), out of its case, stand it up, screw it in the thermometer, and connect the india-rubber tubes carrying the water to the inlet and outlet tubes of the jacket. Turn the milled head, and open and throw

back on to its support the lower prism; see that both glass surfaces are clean (a cloth dipped in alcohol, followed by a dry soft cloth is best for cleaning), and place a drop of the melted fat in the centre of the surface of the lower prism (Fig. 38), and close it.

Arrange the mirror to reflect either day-light or the light of a lamp through the prisms, and observe the point on the scale where the dark shadow comes, focusing the eye-piece if necessary; make two or three observations till the position of the shadow is constant, and read the temperature at the same time. The thermometer may be graduated either in centigrade degrees, or in the normal reading for butter fat at the temperature of the water; if of the former kind, both readings are noted, and the refractometer readings corrected to a standard temperature, by multiplying the difference between the observed temperature and the standard temperature by 0.55, and adding or subtracting the result, as the temperature is higher or lower than the standard, to the refractometer reading. If the thermometer is of the latter type, the observed reading of the refractometer is subtracted from the reading of thermometer, and the result is expressed in degrees less than the standard.

Table VII. gives the standard readings for each five degrees centigrade

TABLE VIII.

Temperature.	Scale Divisions.
25	52.5
30	49.8
35	47.0
40	44.2
45	41.5

The average figure for butter is 46° at 35° C. or a little below, and margarine gives about 54°. Genuine butter sometimes gives a reading higher than 47°, and the limits found are 43.5° to 49°.

A standard solution (*normal flüssigkeit*) is supplied with the instrument, and the scale should be adjusted from time to time with this; a point is marked on the scale where the standard solution should read, and a key is provided for adjusting the scale to this.

Detection of Sesamé Oil.—Add to 10c.c. of the melted butter 0.1c.c. of a 2 per cent. alcoholic solution of furfural, add 1c.c. of strong hydrochloric acid, shake well and add 10c.c. of chloroform. A crimson colouration of the aqueous layer indicates sesamé oil. Sometimes coal-tar colours are added to the butter which give a red colour with hydrochloric acid alone; make sure of the absence of these by testing a little of the butter with hydrochloric acid. If these are present use the fatty acids (p. 67) for the test.

If furfural is not available dissolve 0.1 gramme of sugar in 5c.c. of hydrochloric acid, and shake with 10c.c. of melted fat. This will also give a crimson colour with sesamé oil.

The Application of Analysis to the Solution of Problems. *The Detection of Adulteration.*—The President of the Board of Agriculture, under his powers authorised by Clause 4 of the Sale of Food and Drugs Act, 1899, has laid down the limit of 16 per cent. of water in butter, and any quantity above this amount is presumed to have been added. Occasionally water has been worked into butter to add to its weight, but a more frequent occurrence of excess of water occurs in milk-blended butter; in the latter the "curd" will be in the proportion of 1 part for each 10 parts of water, in the former much less.

A washed butter may be distinguished from an unwashed one by containing less than 1 part of curd to 10 parts of water, usually only about 0.5 part; unwashed butter contains about 1 part; a larger percentage of curd indicates that the butter was churned from very sour cream, and will not keep well, or that it has been adulterated with casein. A higher percentage of casein than 0.5 indicates the presence of added casein; this form of adulteration is not uncommon. The percentage

of salt should not exceed 3 in a mild salt butter, but may go up to 5 per cent. or more in other samples. Irish pickled butters are high in water and in salt.

The Preservatives Committee of the Local Government Board recommended that no preservative except boric acid be allowed in butter, and that only to the extent of 0.5 per cent.; this has not yet been legalised.

The detection of margarine is more difficult; the Butter Regulations Committee of the Board of Agriculture has recommended a limit for the Reichert-Wollny figure of 24 c.c., but their recommendation has not yet been adopted.

This figure, as well as 89 as a superior limit for insoluble fatty acids, 5 per cent. as the minimum for soluble fatty acids, 22.0 per cent. for the potash required for saponification, 0.910 for the density, 42 per cent. for the iodine absorption, and 48° as a maximum Zeiss butyro-refractometer figure, may be taken as indicating the border-line of genuine butters; the appearance on melting, and under polarised light, may be useful as confirmatory, and a reaction for sesamé oil will establish the presence of margarine.

The average figures for butter and margarine are:

TABLE VIII.

	Butter.	Margarine.	Cocoa-nut.
Reichert-Wollny figure	29 cc.	Practically none	7
Insoluble fatty acids .	87.5 %	95.5 %	85
Soluble fatty acids . .	6.0 %	None	?
Potash required . . .	22.7 %	19.5 %	26
Density	0.913	0.902	?
Iodine absorption . .	37	55	9
Refractometer at 35° .	46°	54°	43°

Cocoa-nut oil has a rather different composition from that of margarine; but as it lowers the Reichert-Wollny figure, while at the same time raising the potash absorption, and lowering the insoluble fatty acids, iodine absorption and the refractometer figure, its detection is not difficult.

CHAPTER VI.

THE ANALYSIS OF CHEESE.

Estimation of Water, Ash, and Salt.—Place 2 or 3 grammes of cheese cut into small pieces in a small flat-bottomed basin, and keep in the water-oven for six hours; the drying proceeds better if the basin is inclined so that the fat runs off the drying cheese; weigh and return to the water-oven, and weigh again at intervals of one hour till the loss is less than one milligramme per hour; the loss may be taken as water.

Pour off as much fat as possible, and macerate the residue in hot amyl alcohol; pour off the amyl alcohol as completely as possible, and ignite the residue as in determining the ash of milk (p. 12); to determine the salt make a determination of chlorine as on p. 14; each cubic centimetre of $\frac{N}{10}$ silver nitrate is equal to 0.00585 grammes salt.

Estimation of Fat.—Weigh about 2 grammes of cheese, cut into small pieces, and transfer to a Stokes tube, add 8c.c. of water, and heat gently till the cheese is softened and disintegrated; then add 10c.c. of hydrochloric acid, and treat as in the Werner-Schmid method (p. 30).

Estimation of Total Nitrogen—Weigh about 1 gramme of cheese and treat by the Kjeldahl method (p. 35).

Estimation of Products of Ripening.—Weigh 10 grammes of cheese, place in a small mortar, and add 25c.c. of boiling water; with a pestle grind up the

cheese and water, and pour off the solution through a filter, collecting the filtrate in a 250c.c. flask; repeat the treatment with 25c.c. of boiling water till nine portions have been used; cool the total filtrates, make up to 250c.c., and mix well. Evaporate 50c.c. in a weighed basin, and weigh the solids after drying till the loss is less than 1 milligramme per hour; ignite, and weigh the ash; the weight of the solids less that of the ash represents the products of ripening. The difference between 100, and the sum of the water, fat, ash, and products of ripening, may be taken as unaltered paracasein.

The products of ripening may be differentiated; to 50c.c. of the filtrate add 5c.c. of copper sulphate, and treat as in the Ritthausen method for the estimation of proteids (p. 34); the proteids estimated in this way may be termed primary products of ripening, and the remainder secondary products.

Examination of the Fat.—Dry 25 to 50 grammes of cheese till the fat runs out; extract with ether, and wash the ethereal solution with water in a separating funnel; remove the ethereal layer, and drive off the ether, and dry the fat till the ether is completely removed. Examine the fat as directed for butter fat; usually a Reichert-Wollny figure is required.

The Application of Analysis to the Solution of Problems. *Detection of Adulteration.*—Practically the only adulterations of cheese consist in the removal of fat from the milk before curdling, and the addition of foreign matter.

The removal of fat may be judged if the fat is less than 45 per cent. of the dried cheese, or if the fat is less than six times the total nitrogen, both of which standards lead to practically the same result; a large number of cheeses are made with half-skimmed milk (*e.g.*, the evening's milk is skimmed, and mixed with the fresh morning's milk); these fail to comply with the above standards, and should be sold as half-skim

cheeses; other cheeses are made from skim-milk alone, *e.g.*, Dutch cheese (though cheeses are made in Holland with whole milk also).

The detection of "margarine-cheese" is accomplished by the examination of the fat; practically the same standards as for butter may be used; it must be remembered, however, that during the ripening the fat is slightly attacked, and the percentage of volatile acids may be somewhat lowered by this cause.

Preservatives need not be looked for in cheese.

Application of Analysis to Cheese-making.—*The estimation of the curd* by Lindet's method (p. 38) will give an idea of the value of milk for cheese-making.

A fermentation test is useful; plug a number of clean test-tubes with cotton-wool, and sterilise by heating to 150° C. in an air-bath for half an hour. Place 10c.c. of the milk to be examined in one of these, and keep it at blood-heat for eighteen hours; if the precipitated curd is distended by bubbles of gas the milk will not make good cheese.

The acidity of the milk before renneting should be estimated (p. 16), and that of the whey after the curd is cut, and the whey running from the curd at intervals. As an example of the use of the acidity test, in Cheddar cheese-making the best acidity of the milk for renneting is 22°–24°; the acidity of the whey is less than this, but constantly increases; the whey should be drawn at about 22° acidity, and the curd vatted when the whey draining off has an acidity of about 100°.

The acidity of the curd may also be judged by the hot-iron test; an iron is heated and allowed to cool till it can just be touched with the finger; the acidity of the curd is judged by the length of the string which is formed when the iron is pressed on the curd and withdrawn.

The strength of rennet is determined by weighing out 0.5 gramme of a solid extract, or measuring 5c.c. of a liquid extract, and diluting to 100c.c. 1c.c. of the

solution is added to 100c.c. of separated milk of acidity 20° at a temperature of 35° C.; the temperature is kept at 35°, and the milk slowly stirred with a thermometer till it curdles, which is indicated by the path of the thermometer becoming visible; the time which has elapsed since the addition of the rennet is noted, and the strength of the rennet calculated as parts of rennet which will cause curdling in 40 minutes by the following formulæ:

$$\text{Strength} = \frac{800{,}000}{T} \text{ for solid extracts,}$$

$$\text{or} \quad \frac{80{,}000}{T} \text{ for liquid extracts,}$$

where T = time in minutes.

The curdling should not take less than 5 minutes nor more than 10 minutes, and should this be the case less or more of the rennet solution should be used, and the results increased or decreased proportionately.

TABLE IX

For Correcting Specific Gravity to 60° F.

Temperature Degrees F.	Degrees of Specific Gravity observed.											
	25	26	27	28	29	30	31	32	33	34	35	36
	Specific Gravity corrected to 60° F.											
40	23.5	24.5	25.5	26.4	27.3	28.2	29.1	30.0	31.0	31.9	32.8	33.7
42	23.6	24.6	25.6	26.5	27.5	28.4	29.3	30.2	31.1	32.0	32.9	33.9
44	23.8	24.8	25.8	26.7	27.7	28.6	29.5	30.4	31.3	32.2	33.1	34.1
46	23.9	24.9	25.9	26.8	27.8	28.7	29.6	30.5	31.4	32.4	33.3	34.3
48	24.0	25.0	26.0	26.9	27.9	28.8	29.7	30.6	31.6	32.6	33.5	34.5
50	24.1	25.1	26.1	27.0	28.0	29.0	29.9	30.9	31.8	32.8	33.7	34.7
52	24.3	25.2	26.2	27.2	28.1	29.1	30.1	31.1	32.0	33.0	33.9	34.9
54	24.5	25.4	26.4	27.4	28.4	29.3	30.3	31.3	32.3	33.3	34.2	35.1
56	24.6	25.6	26.6	27.6	28.6	29.6	30.5	31.5	32.5	33.5	34.4	35.4
58	24.8	25.8	26.8	27.8	28.8	29.8	30.8	31.7	32.7	33.7	34.7	35.7
60	**25.0**	**26.0**	**27.0**	**28.0**	**29.0**	**30.0**	**31.0**	**32.0**	**33.0**	**34.0**	**35.0**	**36.0**
62	25.2	26.2	27.3	28.3	29.3	30.3	31.3	32.3	33.3	34.3	35.3	—
64	25.4	26.5	27.5	28.5	29.5	30.5	31.5	32.6	33.6	34.6	35.6	—
66	25.6	26.7	27.7	28.7	29.8	30.8	31.8	32.9	33.9	34.9	35.9	—
68	25.9	27.0	28.0	29.0	30.1	31.1	32.1	33.2	34.2	35.2	36.2	—
70	26.1	27.2	28.2	29.2	30.3	31.3	32.4	33.4	34.5	35.5	36.5	—
72	26.4	27.4	28.4	29.5	30.5	31.6	32.6	33.7	34.7	35.8	—	—
74	26.6	27.7	28.7	29.7	30.8	31.9	32.9	34.0	35.0	36.1	—	—
76	26.9	27.9	28.9	29.9	31.0	32.2	33.3	34.4	35.4	36.5	—	—
78	27.2	28.2	29.2	30.3	31.4	32.5	33.6	34.7	35.8	36.9	—	—
80	27.4	28.4	29.5	30.6	31.7	32.8	33.9	35.0	36.1	—	—	—

TABLE X.

For Calculating Solids not Fat from Fat and Specific Gravity.

Correction.	* Specific Gravity (degrees).							
* −1.0	25.0	25.5	26.0	26.5	27.0	27.5	28.0	28.5
	29.0	**29.5**	**30.0**	**30.5**	**31.0**	**31.5**	**32.0**	**32.5**
+1.0	33.0	33.5	34.0	34.5	35.0	35.5	36.0	36.5
Fat.	Solids not Fat.							
0.25	7.45	7.55	7.70	7.80	7.95	8.05	8.20	8.30
0.5	7.50	7.60	7.75	7.85	8.00	8.10	8.25	8.35
0.75	7.55	7.65	7.80	7.90	8.05	8.15	8.30	8.40
1.0	7.60	7.70	7.85	7.95	8.10	8.20	8.35	8.45
1.25	7.65	7.75	7.90	8.00	8.15	8.25	8.40	8.50
1.5	7.70	7.80	7.95	8.05	8.20	8.30	8.45	8.55
1.75	7.75	7.85	8.00	8.10	8.25	8.35	8.50	8.60
2.0	7.80	7.90	8.05	8.15	8.30	8.40	8.55	8.65
2.75	7.85	7.95	8.10	8.20	8.35	8.45	8.60	8.70
2.5	7.90	8.00	8.15	8.25	8.40	8.50	8.65	8.75
2.75	7.95	8.05	8.20	8.30	8.45	8.55	8.70	8.80
3.0	8.00	8.10	8.25	8.35	8.50	8.60	8.75	8.85
3.25	8.05	8.15	8.30	8.40	8.55	8.65	8.80	8.90
3.5	8.10	8.20	8.35	8.45	8.60	8.70	8.85	8.95
3.75	8.15	8.25	8.40	8.50	8.65	8.75	8.90	9.00
4.0	8.20	8.30	8.45	8.55	8.70	8.80	8.95	9.05
4.25	8.25	8.35	8.50	8.60	8.75	8.85	9.00	9.10
4.5	8.30	8.40	8.55	8.65	8.80	8.90	9.05	9.15
4.75	8.35	8.45	8.60	8.70	8.85	8.95	9.10	9.20
5.0	8.40	8.50	8.65	8.75	8.90	9.00	9.15	9.25
†Change at	0.2	0.1	0.2	0.1	0.2	0.1	0.2	0.1

* The solids not fat are correct for the middle line of specific gravity; if the specific gravity falls in the top line subtract 1 from the solids not fat; thus 26.0 sp. gr. and 3.0% fat give 7.25% solids not fat; if the specific gravity falls in the bottom line, add 1 to the solids not fat; thus 34.0 sp. gr. and 3.0% fat give 9.25% solids not fat.

† The last line indicates where the change of solids not fat takes place; in a column with 0.2 at the bottom use the column itself for the percentage of fat given, and 0.05, 0.10, and 0.15 above, but use the figure immediately below for 0.2 above; thus 30.0 sp. gr. and 3.1% fat give 8.25% solids not fat, but 30.0 sp. gr. and 3.2% fat give 8.30% solids not fat; in a column with 0.1 at the bottom, use the column itself for the percentage of fat given and 0.05 above, but change to the figure immediately below for 0.1 or more above; thus 30.5 sp. gr. and 3.30% fat give 8.40% solids not fat, but 30.5 sp. gr. and 3.40% fat give 8.45% solids not fat.

TABLE XI.

For Calculating Fat from Total Solids and Specific Gravity.

	Specific Gravity Observed.									
	A25.0 B29.0 C33.0	25.5 29.5 33.5	26.0 30.0 34.0	26.5 30.5 34.5		A27.0 B31.0 C35.0	27.5 31.5 35.5	28.0 32.0 36.0	28.5 32.5 36.5	
Total Solids A	Fat				Total Solids B	Fat				Total Solids C
7.00	0.50	0.40	0.30	0.20	8.0	0.10	—	—	—	9.00
7.25	0.70	0.60	0.50	0 40	8.25	0.30	0.20	0.10	—	9.25
7.50	0.90	0.80	0.70	0.60	8.5	0.50	0.40	0.30	0.20	9.50
7.75	1.15	1.05	0.90	0.80	8.75	0.70	0.60	0.50	0.40	9.75
8.00	1.35	1.25	1.15	1.05	9.00	0.90	0.80	0.70	0.60	10.00
8.25	1.55	1.45	1.35	1.25	9.25	1.15	1.05	0.90	0.80	10.25
8.50	1 75	1.65	1.55	1.45	9.50	1.35	1.25	1.15	1.05	10.50
8.75	1.95	1.85	1.75	1.65	9.75	1.55	1.45	1.35	1.25	10.75
9.00	2.15	2.05	1.95	1.85	10.00	1.75	1.65	1.55	1.45	11.00
9.25	2.40	2.30	2.15	2.05	10.25	1.95	1.85	1.75	1.65	11.25
9.50	2.60	2.50	2.40	2.30	10.50	2.15	2.05	1.95	1.85	11.50
9.75	2.80	2.70	2.60	2.50	10.75	2.40	2.30	2.15	2.01	11.75
10.00	3.00	2.90	2.80	2.70	11 00	2.60	2.50	2.40	2.30	12.00
10.25	3 20	3.10	3.00	2.90	11.25	2.80	2.70	2.60	2.50	12.25
10.50	3.40	3.30	3.20	3.10	11.50	3.00	2.90	2.80	2.70	12.50
10.75	3.65	3.55	3.40	3.30	11.75	3.20	3.10	3.00	2.90	12.75
11.00	3.85	3.75	3.65	3.55	12.00	3.40	3.30	3.20	3.10	13.00
11.25	4.05	3.95	3.85	3.75	12.25	3.65	3.55	3.40	3.30	13.25
11.50	4 25	4.15	4.05	3.95	12.50	3.85	3.75	3.65	3.55	13.50
11.75	4.45	4.35	4.25	4.15	12.75	4.05	3.95	3.85	3.75	13.75
12.00	4.65	4.55	4.45	4.35	13.00	4.25	4.15	4.05	3.95	14.00
12.25	4.90	4.80	4.65	4.55	13.25	4.45	4.35	4.25	4.15	14.25
12.50	5.10	5.00	4.90	4.80	13.50	4.65	4.55	4.45	4.35	14.50
12.75	5.30	5.20	5.10	5.00	13.75	4.90	4.80	4.65	4.55	14.75
13.00	5.50	5.40	5.30	5.20	14.00	5.10	5.00	4.90	4.80	15.00

If the total solids do not exactly agree with a figure in the Table add the excess of total solids to the fat; thus, given 30.5 sp. gr. and 11.8% total solids, add 0.05 to fat corresponding with 11.75% total solids = 3.35% fat.

If the specific gravity lies in line marked A use the total solids column marked A; if in B use total solids column B; if in C use total solids column C.

APPENDIX

PREPARATION OF STANDARD SOLUTIONS.

As the preparation of a standard solution is rarely, if ever, undertaken while an analysis is being performed, and as also it is tedious to wade through a description of a method, when seeking only for the quantities to be weighed or measured for the solution, details of the preparation have been removed from the text; the arrangement is alphabetical.

Alcoholic Soda Solution.— $\frac{N}{2}$ approximately. Add 25c.c. of a 50 per cent. caustic soda solution (see p. 84) to 1 litre of alcohol (sp. gr. 0.830) and mix well; allow the solution to stand a day, filter into a dry stoppered bottle, and titrate (see Titration) against $\frac{N}{2}$ hydrochloric acid (see p. 83) to ascertain strength. As this solution loses strength on keeping, it must be titrated each time it is used.

Amyl Alcohol (for Gerber test) should conform to the following: Density, 0.8145 to 0.816 at 15.5° C. (Water at 15.5° C. = 1.) Boiling-point: should not begin to boil below 124° C.; not more than 5c.c. should distil at 127.5° C. when 25c.c. are boiled in a 100c.c. flask, and the boiling-point should not rise above 130.5° C.; 10c.c. should completely mix with 10c.c. of hydrochloric acid (sp. gr. 1.17) and the addition of 1.5c.c. of water should produce a permanent turbidity. 2c.c. treated in the Gerber bottle with

10c.c. water and 10c.c. sulphuric acid should yield no layer of "fat."

Baryta Solution.—Dissolve 33–35 grammes of barium hydrate in 1 litre of water; allow to settle, and store in a bottle fitted with a soda lime tube to prevent access of CO_2. Check the strength by titration (see p. 85).

Caustic Soda. See Soda.

Fehling's Solution.—Copper sulphate solution: Dissolve 34.639 grammes of crystallised copper sulphate in water, and make up to 500c.c. Alkaline tartrate solution: Dissolve 173 grammes of pure sodium potassium tartrate (Rochelle Salt), and 51 to 55 grammes of sodium hydroxide of good quality in water, and make up to 500c.c.

Hydrochloric Acid, $\frac{N}{2}$.—Dilute 50c.c. strong hydrochloric acid to 1 litre; ignite a quantity of pure sodium bi-carbonate at a dull red heat, and cool in a desiccator; weigh (accurately to 1 milligramme) portions of this, of about 1 gramme each, dissolve in water, add a little methyl orange, and run in the hydrochloric acid solution from a burette till the sodium carbonate solution turns pink; the solution should be covered as much as possible with a watch-glass to prevent loss by spirting as the CO_2 is given off. Wash the watch-glass into the solution, and boil it, and continue the titration till the pink colour is permanent on boiling. It is well to place a beaker containing an equal bulk of distilled water, and the same volume of methyl orange solution, and one drop (0.05c.c.) of hydrochloric acid, and titrate till the sodium carbonate solution has the same colour, and subtract 0.05c.c. from the quantity of acid used. If the solution is $\frac{N}{2}$, 20c.c. should be required for each 0.53 grammes of sodium carbonate; if this quantity is not used dilute in the proportion of the quantity used to 20c.c., or use the solution as it is, and multiply by 20 divided by the quantity used to obtain its value in

terms of $\frac{N}{2}$ acid. $\frac{N}{10}$ solution is made from $\frac{N}{2}$ solution by diluting 200c.c. to 1 litre.

Iodine Solution (v. Hubl). *Iodine Solution.*—Dissolve 25 grammes of iodine in 500c.c. of 95 per cent. alcohol (*not* methylated).

Mercuric Chloride Solution.—Dissolve 30 grammes of mercuric chloride in 500c.c. of 95 per cent. alcohol (*not* methylated). Equal volumes of these two solutions are mixed at least 24 hours before use.

Magnesia Mixture.—Mix 60 grammes magnesium chloride ($MgCl_2 6OH_2$), 145 grammes ammonium chloride, 600c.c. water, and 300c.c. ammonia solution (sp. gr. 0.880).

Mercuric Nitrate Solution (Wiley).—Dissolve mercury in twice its weight of nitric acid (sp. gr. 1.42), and, after solution, add an equal bulk of water.

Nessler Solution.—Dissolve 35 grammes of potassium iodide in 100c.c. of water; set aside a few c.c. of this solution, and add to the remainder a saturated solution of mercuric chloride till a permanent red precipitate is formed; add the small portion set aside, and cautiously drop in mercuric chloride solution, till a faint permanent precipitate is left. Dissolve 160 grammes of potassium hydroxide in water; *cool* the solution, and add it to the potassium mercuric iodide solution. Make up to 1 litre, add a little mercuric chloride solution and allow to stand till clear.

Silver Nitrate, $\frac{N}{10}$.—Dissolve 17.000 grammes of silver nitrate in 1 litre of water.

Soda Solution, 50 per cent.—Dissolve 250 grammes of caustic soda (purified by alcohol) in 250c.c. of water; allow to stand till clear, and store in the apparatus described on p. 64.

$\frac{N}{2}$.—Dilute 25 c.c. of the 50 per cent. solution to 1 litre. Check the strength by titration (see p. 85).

$\frac{N}{10}$.—Dilute 5c.c. of the 50 per cent. solution to 1 litre. Check the strength by titration.

Strontia Solution.—Dissolve 28–30 grammes of strontium hydrate in 1 litre of water. Treat as baryta solution (p. 83).

Sulphuric Acid. *For Gerber Process.*—Should contain 90–91 per cent. H_2SO_4. Acid of specific gravity 1.820 to 1.825 fulfils this requirement.

For Reichert Process.—Dilute 25c.c. strong sulphuric acid to 1 litre of water; 2c.c. of 50 per cent. soda solution should neutralise about 35c.c. of this solution; adjust the strength, if necessary, by adding acid or water.

$\frac{N}{10}$.—Dilute 3c.c. of strong sulphuric acid to 1 litre. Check the strength as for hydrochloric acid (0.2 grammes only of the sodium carbonate is weighed for $\frac{N}{10}$ acid, and 100c.c. of $\frac{N}{10}$ acid should be required for 0.53 grammes sodium carbonate).

Thiosulphate Solution.—Dissolve 25 grammes of pure sodium thiosulphate and 1 gramme of salicylic acid in 1 litre of water. Allow to stand for a few days and filter. Weigh accurately about 0.25 gramme of iodine in a small stoppered flask, add 2 grammes potassium iodide and 2c.c. of water, and shake gently till the iodine is dissolved. Dilute with water and transfer to a larger vessel, and run in the thiosulphate solution from a burette till the yellow colour just disappears. Repeat this two or three times, and from the mean results calculate the weight of iodine equivalent to 1c.c.

Titration.—Take two burettes, fill one with the alkaline solution, and the other with hydrochloric acid solution of corresponding strength. Measure 25c.c. of the acid solution, add a few drops of phenolphthalein solution (or cochineal solution if this is used in the experiment for which the alkali is to be employed), and run in the alkali till a pink colour (violet with cochineal) is produced. Note the volume of alkali used. Repeat this experiment two or three times, and from the mean of the results calculate the ratio of the alkali solution to

the acid solution. Thus, if 24.2, 24.15, and 24.2c.c. of $\frac{N}{2}$ soda were used for 25c.c. acid the ratio is $\frac{25}{24.17} = 1.034$ or 1 c.c. of soda = 1.034c.c. acid.

If the acid is strictly $\frac{N}{2}$ the soda is $\frac{N}{2} \times 1.034$; if the acid is, for instance, $\frac{N}{2} \times 1.011$, the soda is then $\frac{N}{2} \times 1.034 \times 1.011 = \frac{N}{2} \times 1.0454$.

The soda may be diluted in the ratio of 1000 parts to 1045.4 parts, *i.e.*, 45.4 c.c. of water are added to each litre, but it is preferable to use the solution as it is, and multiply the results by the factor.

INDEX

ABSORPTION, iodine, 69
potash, 67
Abstraction of fat, 49
Acidity, estimation of, 16
of curd, 77
Acidobutyrometer, 17
Adams' method for fat estimation, 28, 43, 46
Adulteration, detection of
in butter, 73
in cheese, 76
in cream, 58
in milk, 49
in skim milk, 59
Albumin, 1, 2
estimation of, 35, 37
Alcohol in sour milk, estimation of, 47
amyl, 17, 82
Alcoholic soda solution, 82
Alkalinity of ash, estimation of, 13
Ammonia in sour milk, estimation of, 48
Amyl alcohol (for Gerber Test), 17, 82
Annatto, detection of, 54
Ash, estimation of, in milk, 12, 13
in cheese, 75

BABCOCK'S method of total solid estimation, 12
Blood in milk, detection of, 54
Boiled milk, test for, 52
Boric acid, in butter, 61
in milk, 51
Butter, analysis of, 60–74
composition of, 4, 74
Butter, detection of adulteration of, 73
examination by polariscope, 68
preservatives in, 61, 74
-milk, 4, 45

CANE sugar, estimation of, 46, 52
Casein, 1, 2
estimation of, 35, 37
in butter, 61
Cause of dirty milk, 58
high colour in milk, 53
poor milk, 52
sour milk, 54
sweet milk, 52
unusual taste in milk, 57
Caustic soda solution (*see* Soda)
Centrifuges, 18, 53
Cheese, 3
analysis of, 75–78
composition of, 4
-making, application of analysis to, 77
products of ripening of, 75
Chlorine, estimation of, 13
Coal-tar dyes, detection of, 54
Cocoa-nut oil, detection of, 74
Colour, cause of, in milk, 53
Composition of butter, 4, 74
cocoa-nut oil, 74
fat, 62
margarine, 63
milk, 3, 4, 49
morning and evening milk, 3
products, 4
Condensed milk, 45
Cream, 3
analysis of, 24, 43

Cream, calculation of fat in, 44
composition of, 4
detection of abstraction of, 50
detection of adulteration of, 58
Curd, 2, 3, 54
composition of, 4
estimation of,
in butter, 60
in cheese, 77
in milk, 38
Curdling, cause of, 54
Custard, cause of curdling in, 55

DENSITY of butter, 68
(*see* also Specific gravity)
Detection of adulteration of
butter, 73
cheese, 76
cream, 58
milk, 49
of preservatives in
butter, 61
cream, 59
milk, 51
Dirty milk, cause of, 58
Dyes, in milk, 54

EXAMINATION of fat in
butter, 62
under polarised light, 68
cream, 59
cheese, 75
milk, 59

FAT globules, 1
gravimetric estimation of, 27–30
in butter, 62
in cheese, 75
in cream, 44
in sour milk, 46
preparation of, for analysis, 64
relation between specific gravity, solids not fat, and, 39
standard for, 50
volumetric estimation of, 17–27
Fatty acids, soluble and insoluble, estimation of, 66
volatile, estimation of, 64
Fermentation test (cheese), 77
Fluorides, detection of, 62
Formaldehyde, detection of, 51

GELATINE, detection of, 58
Gerber's method, for fat estimation, 17–27, 43
Gottlieb's method, for fat estimation, 30, 43
Gravimetric estimation of specific gravity, 10

HEATED milk, detection of, 52
High colour, cause of, in milk, 52
Hydrochloric acid, 83
Hydrogen peroxide, detection of, 51

INSOLUBLE ash, estimation of, 13
fatty acids, estimation of, 66
Iodine absorption, 69
solution, 84

KJELDAHL'S method of estimating nitrogen, 35
Kieselguhr, 29

LACTIC acid, 2, 54
estimation of, 16
Lactometers, 6 *et seq.*
Leffmann and Beam's method
of fat estimation, 17
of butter analysis, 66
Lime, 2
detection of added, 59
estimation of, 13
Lindet's method for curd, 38, 77

MACHINE-SKIMMED milk (*see* Separated milk)
Magnesia mixture, 84
Margarine, composition of, 63
detection of, 63, 69, 74
-cheese, detection of, 77

Mean molecular weight of fatty acids, 66
Measuring apparatus for milk, 18
Mercuric nitrate solution, 84
Micro-organisms, action of, on milk, 2, 54
Microscope, use of, 55, 57
Milk, composition of, 3, 4, 49
measurement of, 19
morning and evening, composition of, 3
scale, 40
sour, analysis of, 6, 46
standards for, 50
sugar, 1
estimation of, 30–34, 44

NESSLER solution, 84
Nitrogen, estimation of, in milk, 35
estimation of, in cheese, 75

PARA-CASEIN, 2
Phosphoric acid, estimation of, 13, 38
Polarimetric estimation of milk sugar, 30, 44
Polarised light, examination of butter under, 68
Poor milk, cause of, 52
Preparation of fat for analysis, 64
samples of milk, 5
Preservatives, detection of,
in butter, 61
in cream, 59
in milk, 51
Products of ripening of cheese, 75
Proteids, 2
estimation of, 34–39, 45

REFRACTIVE index of butter, 69
Reichert - Wollny method for estimation of volatile fatty acids, 64
Rennet, 2, 3
detection of, 59
estimation of strength of, 77
Ripening of cheese, products of, 75
Rise of specific gravity of milk on standing, 11
Ritthausen's method for fat estimation, 29, 35, 43
method for proteids, 34

SALICYLIC acid, detection of, 51
Salt, estimation of, in butter, 60
in cheese, 75
Separated milk, 3
adulteration of, 59
analysis of, 43
standard for, 58
Sesamé oil, 73, 74
Silver nitrate, 84
Skim milk (*see* Separated milk)
Skimming, detection of, 50
Soda solution, 84
Sodium thiosulphate solution, 85
Solids, estimation of total, 11
not fat, estimation of, 39
relation between fat and specific gravity, 39
standards for, 50
Soluble ash, estimation of, 13
fatty acids, estimation of, 66
Solution of problems, 49–59, 73–74, 76
Sour milk, analysis of, 6, 46
cause of, 2, 54
Soxhlet extractor, 29
Specific gravity, correction for temperature, 41, 79
estimation by lactometer, 6
gravimetric estimation, 10
of sour milk, 46
relation between fat, solids not fat, and, 39
Specific gravity, rise of, in milk, on standing, 11
Sprengel tube, 10
Standards for butter, 74
milk, 50
skim milk, 58
Starch, detection of, 58

Sterilised milk, 45
Stokes method for total solids estimation, 12
tube, 30
Storch's method for fat estimation, 29, 43
Strontia solution, 85
Sugar, cane, estimation of, 46, 52
milk- (*see* Milk-sugar)
Sulphur, estimation of, 38
Sweet milk, cause of, 52

TASTE, cause of unusual, in milk, 57
Thermo-lactometer, 8
Thiosulphate solution, 85
Titration, 85
Total solids, estimation of, 11
by calculation, 39, 81
Total solids, in cream, 43
Total solids, in sour milk, 46

UNUSUAL taste in milk, cause of, 57

VEGETABLE matter in milk, 56, 58
Viscogen, detection of, 59
Volatile acid in sour milk, 47
fatty acids, estimation of, 64

WATER in butter, estimation of, 60
cheese, 75
standard for, 73
milk, detection of, 49
Werner-Schmid, method of fat estimation, 30, 46
Whey, 2, 45

ZEISS Butyro-refractometer, 69

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BAIRD & TATLOCK (LONDON) LTD.'S LIST OF APPARATUS AND CHEMICALS REQUIRED FOR MILK ANALYSIS, &c.

As mentioned in the "Laboratory Book of Dairy Analysis," by H. DROOP RICHMOND, F.I.C.

ALL the apparatus detailed below are required for a full equipment; the quantity will vary according to requirements:

THE ANALYSIS OF MILK.

Sampling.

Page	*Item No.*	
5	1	Sample bottles, oval shape, with corks, 4 oz.
6	2	Beakers with spout, No. 5.
6	3	Brush, fine wire, for mixing.

Specific Gravity.

7	4	Lactometer, ordinary pattern, paper scale.
7	5	Lactometer, Veith's pattern, 25–35.
7	6	Lactometer, Soxhlet's pattern, about 13 in. long.
7	7	Lactometer jar, zinc, 4 in. by $1\frac{3}{4}$ in., with spout.
8	8	Thermo-lactometer, Quevenne's.
10	9	Beaker flasks, 200c.c.
10	10	Sprengel tube, 10c.c.

Estimation of Total Solids.

11	11	Platinum basin, flat, shallow, with spout, $2\frac{3}{4}$ in.
11	12	Porcelain capsule, glazed all over.
11	13	Pipette for milk, 5c.c., with mark.
11	14	Pipette to hold 5 grammes of milk.

Estimation of Ash, &c.

11	15	Water-bath to take 6 porcelain capsules.
12	16	Desiccator, Scheibler's pattern, 6 in.
12	17	Balance, Bunge's 200 grammes sensibility, $\frac{1}{10}$th mgm. on plate-glass sole.
12	18	Set of gilt weights for above, 50 to .001 grammes.
12	19	Muffle furnace, No. 461.
12	20	Fireclay muffle for ditto.

Page	*Item No.*	
12	21	Platinum wire, 6 in. long.
12	22	Tripod, triangular, 8 in. high.
12	23	Bunsen burner, ½ in.
12	24	India-rubber tubing for above, heavy walls.
12	25	Pipe-clay triangle, Clowes' improved form.
13	26	Filter paper, C.S. and S. 589, black band.
15	27	Porcelain basin, round bottomed, with spout, diameter 3½ in.

Estimation of Acidity.

16	28	Acidimeter for testing acidity, consisting of special burette, stand, and dropping bottle.

Estimation of Fat.

17	29	Gerber's butyrometer for 4 samples, with accessories as follows: 4 bottles; 1 pipette, 3c.c. for cream; 1 ,, 10c.c. 100 divs. for water; 1 ,, 11c.c. for milk; 1 ,, 10c.c. for acid; 1 ,, 1c.c. for amyl alcohol.
17	30	Leffman-Beam centrifugal machine, for 4 samples, with accessories as follows: 4 bottles; 1 pipette, 3c.c.; 1 ,, 9c.c.; 1 ,, 15c.c.
20	31	Richmond's shaking stand for 8 Gerber's tubes.
25	32	Pair of tared tin dishes, 2 in. by 1⅝ in., for weighing cream.

Gravimetric Estimation of Fat.

28	33	Adams' fat-free paper, in strips.
30	34	Werner-Schmid tube.
29	35	Fat extraction thimble.
29	36	Glass mortar, 3 in. diameter, with pestle.
35	37	Funnel, 2½ in.
35	38	Filter paper, C.S. and S 595, to suit.
35	39	Beakers, No. 3, with spout.
35	40	6 glass stirring rods, 6 in. long.
35	41	Conical flasks, 6 oz.

Page	Item No.	
29	42	Soxhlet's Fat Extraction Apparatus as follows : 1 water-bath for 3 apparatus on stand ; 1 set Bunsen's for ditto ; 3 fat extractors, 60 grammes ; 3 flasks with short necks, 4 oz. ; 3 condensers with india-rubber caps ; 1 stand with three-armed clamp for supporting condensers.
30	43	Stokes tube.
30	44	Burette, 100c.c. $\frac{1}{10}$ths, with stopcock. Mahogany stand for ditto.
		Estimation of Milk Sugar.
31	45	Mitscherlich's polariscope, with 100 and 200 mm. tube and burner.
33	46	Hard glass tubes, 10 × 1c.m., with one end drawn down.
31	47	Flasks, with mark 100c.c., unstoppered.
31	48	Policemen, with india-rubber tops, for stirring.
33	49	Filter pump glass.
33	50	Glass tubing, assorted.
33	51	Wash bottles, fitted with cork and tubes, 1 litre.
		Estimation of Proteids.
34	52	Gooch's porcelain crucible.
34	53	Asbestos fibre, for use with above.
		Estimation of Casein and Albumin.
35	54	Porcelain crucibles, No. 1, with lids.
35	55	Crucible tongs, gun-metal, with platinum tips.
		Estimation of Nitrogen.
35	56	Kjeldahl's apparatus, with litre copper flask, Liebig's condenser, &c.
35	57	Flask, 200c.c., hard glass, round bottom.
40	58	Richmond's improved milk slide rule.
		Analysis of Liquid Milk-Products.
46	59	Pipette, 5c.c., short form with india-rubber tube at top.
47	60	Flask, with side tube, 250c.c.
47	61	Liebig's condenser, for use with ditto.

Page	Item No.	
48	62	Nessler tubes, marked at 50c.c.

The Application of Analysis to the Solutions of Problems.

52	63	Test-tubes, $6 \times \frac{5}{8}$ in.
53	64	High-speed centrifuge.
58	65	Microscope, with $\frac{2}{3}$ in. and $\frac{1}{6}$ in. objective, one eyepiece, and substage condenser and polariscope fitted.
15, 61, 85	66	2 burettes, 50c.c. $\frac{1}{10}$ths.
18, 61, 85	67	Stand, double, for ditto.
15, 61, 85	68	6 dropping bottles, with grooved stopper, 30c.c.

THE ANALYSIS OF BUTTER.

Estimation of Water, &c.

60	69	Sand-bath, 6 in.
60	70	1 round tripod for ditto.
60	71	1 Bunsen burner for ditto.
60	72	Porcelain basins, No. 1.
60	73	Glass rod for ditto.
62	74	Glass plates, $2\frac{1}{2} \times 2\frac{1}{2}$ in.
62	75	Set of pipettes, with mark 5, 10, 20 and 25 c.c.

Estimation of the Volatile Fatty Acids.

64	76	Caustic soda apparatus.
66	77	Reichert-Wollny apparatus.
67	78	Flask, CO_2, 6 oz.
66	79	Liebig's condenser for ditto.
67	80	Porcelain basin, 6 in. diameter.
68	81	Microscope slides, 3×1 in.
68	82	Microscope cover glasses, $\frac{5}{8}$ in. square, No. 2.
69	83	Bottles, 4 oz. N.M., flat stoppered.

Determination of Refractive Index.

70	84	Zeiss' butyro-refractometer.

Page	*Item No.*	
		THE ANALYSIS OF CHEESE.
75	85	Porcelain capsules, glazed all over.
76	86	Flask, 250c.c. with mark, stoppered.
76	87	Separators, 500c.c. cylindrical, with stopper and stopcock.
77	88	Hot air-bath, 7 × 7 in., on stand.
78	89	Thermometer, engraved up to 110° C.
77	90	Incubator, B and T's form, slag wool lined, with capsule regulating at 37° C., complete with thermometer.
77	91	Pair watch glasses with binder.
77	92	100c.c. flask with mark.

CHEMICALS AND STANDARD SOLUTIONS

THE quantities detailed below are necessary for a large laboratory. They may be reduced in proportion when only a small outfit is required:

1 lb. acetic acid B.P.
4 oz. phenolphthalein indicator.
1 litre sulphuric acid $\frac{N}{10}$ solution.
4 oz. potassium chromate indicator.
1 litre silver nitrate $\frac{N}{10}$ solution.
1 w. qt. hydrochloric acid, pure.
1 w. qt. ammonia, .880.
½ lb. ammonium oxalate, pure.
½ lb. ammonium carbonate, pure.
½ lb. magnesia mixture.
1 litre caustic soda $\frac{N}{10}$ solution.
1 lb. Kieselguhr special for filtering.
1 w. qt. amylic alcohol } for Leffmann-Beam
1 carboy sulphuric acid } process.
1 w. qt. petroleum ether. }
5 lb. caustic soda, pure. }
1 w. qt. sulphuric acid free N. } for Kjeldahl's
1 lb. mercury redistilled. } process.
2 lb. potassium bisulphate. }
1 lb. sodium sulphide pure reagent. }
4 oz. cochineal indicator. }